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INVESTIGATIONS LEADING TO THE DEVELOPMENT
OF A PRIMARY ZINC-SILVER OXIDE BATTERY
OF IMPROVED PERFORMANCE CHARACTERISTICS

SUMMARY REPORT NO. 1

(covering a six-month period)

Contract No. NAS 8-5493

Control Number TP3-83728 (1F)

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GEORGE C. MARSHALL SPACE FLIGHT CENTER
Huntsville, Alabama

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I. PURPOSE

The purpose of this contract is to generate design data making possible the construction of a reliable primary zinc-silver oxide battery of improved activated charge retention characteristics, greater voltage control, high energy density, increased temperature stability, and reduced gassing characteristics.

II. ABSTRACT

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Separator materials have been evaluated with respect to resistance to attack by potassium hydroxide, electrical resistance, speed of wetting, and electrolyte retention. Methods of separator testing and quantitative results are presented.

A series of cells were designed for testing to establish an initial point of reference. The control, or "standard" cell, was revealed not to be the optimum design for a long stand application. It is disclosed that under laboratory conditions, several separator combinations may allow high retention of charge at +70° F.

Several variables have been organized in a "fractional factorial" experiment, which has been initiated. Initial results are presented relative to this experiment design program involving a stand temperature at +130° F.

Author

III. FACTUAL DATA AND DISCUSSION

A. Introduction

1. Outline of Planned Investigation

The following outline of planned investigations has been set forth and serves as a broad basis for fundamental studies relative to the primary zinc-silver oxide system. A master schedule, contained in the Appendix, indicates the planned allotment of time for each phase of study and the progress being attained.

I. EVALUATION OF SEPARATION MATERIALS

A. Types

1. Open
 - a. Webril
 - b. Viskon (R-35-D and R-75-D)
 - c. Nylon (woven and non-woven)
 - d. Polyethylene
 - e. Polypropylene
 - f. Polyvinyl Chloride
 - g. Others
2. Membranes
 - a. Cellulosic
 - 1) 133 Visking
 - 2) Fibrous Visking
 - 3) 300 Cellophane
 - 4) 600 Cellophane
 - b. Inert
 - 1) Permion
 - 2) Polypor (now Acropor)
3. Special Treatments
 - a. PVC dip
 - b. PVA dip

B. Properties for Investigation

1. Resistivity, ohm-cm²
2. Speed of wetting
3. Ability to retard silver migration
4. Retention-of-charge characteristics as a function of temperature
5. Absorbency

II. ACTIVE MATERIAL FORMATION

A. Positive Material

1. Effect of apparent density
2. Effect of formation current density on subsequent capacity efficiency
3. Effect of electrolyte concentration on formation and capacity efficiency

4. Treeing and migration of silver as affected by
 - a. Temperature
 - b. Electrolyte Concentration
 - c. Apparent density of divalent silver oxide
5. Efficiency of charge studies
6. Half-cell studies

B. Negative Materials

1. Efficiency of charge studies
2. Treeing and migration of zinc
 - a. As affected by temperature
 - b. As affected by KOH concentration
 - c. As affected by apparent density
3. Comparison of spongy and metallic zinc
4. Half-cell studies

III. ELECTROLYTE

A. Effect of Concentration

1. Solubility of zinc
2. Solubility of divalent silver oxide
3. Electrolyte resistivity

B. Additives

1. Zinc Oxide
2. Halide salts
3. Carboxymethyl Cellulose
4. Starch
5. Lithium Hydroxide
6. Lignin
7. Others

IV. CELL AND BATTERY CONSTRUCTION

A. Electrolyte Retention

1. Vent plugs
2. Head room
3. Separation

B. Voltage Regulation

C. High rate capabilities

D. Energy Density

E. Thermal Characteristics

V. REACTION MECHANISMS

A. Gassing Rates

1. Effect of additives
2. Metallic as compared to spongy zinc
3. Treated spongy zinc
4. Electrolyte concentration
5. Electrolyte additives
6. Effect of grid material

B. Theoretical Investigation

1. Reaction Mechanisms of Gassing
 - a. Gassing at positives
 - b. Gassing at negatives
2. Establish theoretical hypothesis for preventing gassing

VI. RELIABILITY EVALUATION

A. Failure Effects Analysis

B. Drift Analysis on Parts

C. Stress Analysis and Reliability Estimation for Each Part

2. Goals and Determining Factors Relative to These Goals

Certain major tentative goals have been set forth in accordance with Contract No. NAS 8-5493. These are listed below:

- a. Plateau voltage of 1.40 volts per cell
- b. Maximum possible capacity when discharged at the 20-minute rate
- c. Good voltage control
- d. High energy density
- e. Minimum of 30 days activated stand
- f. Low gassing as required for space applications.

The following is a discussion of several basic concepts involved in attaining these goals.

All other factors remaining constant, plateau voltage is essentially an inverse linear function of current density in amperes per square inch of active material surface area as illustrated by Figure No. 1.

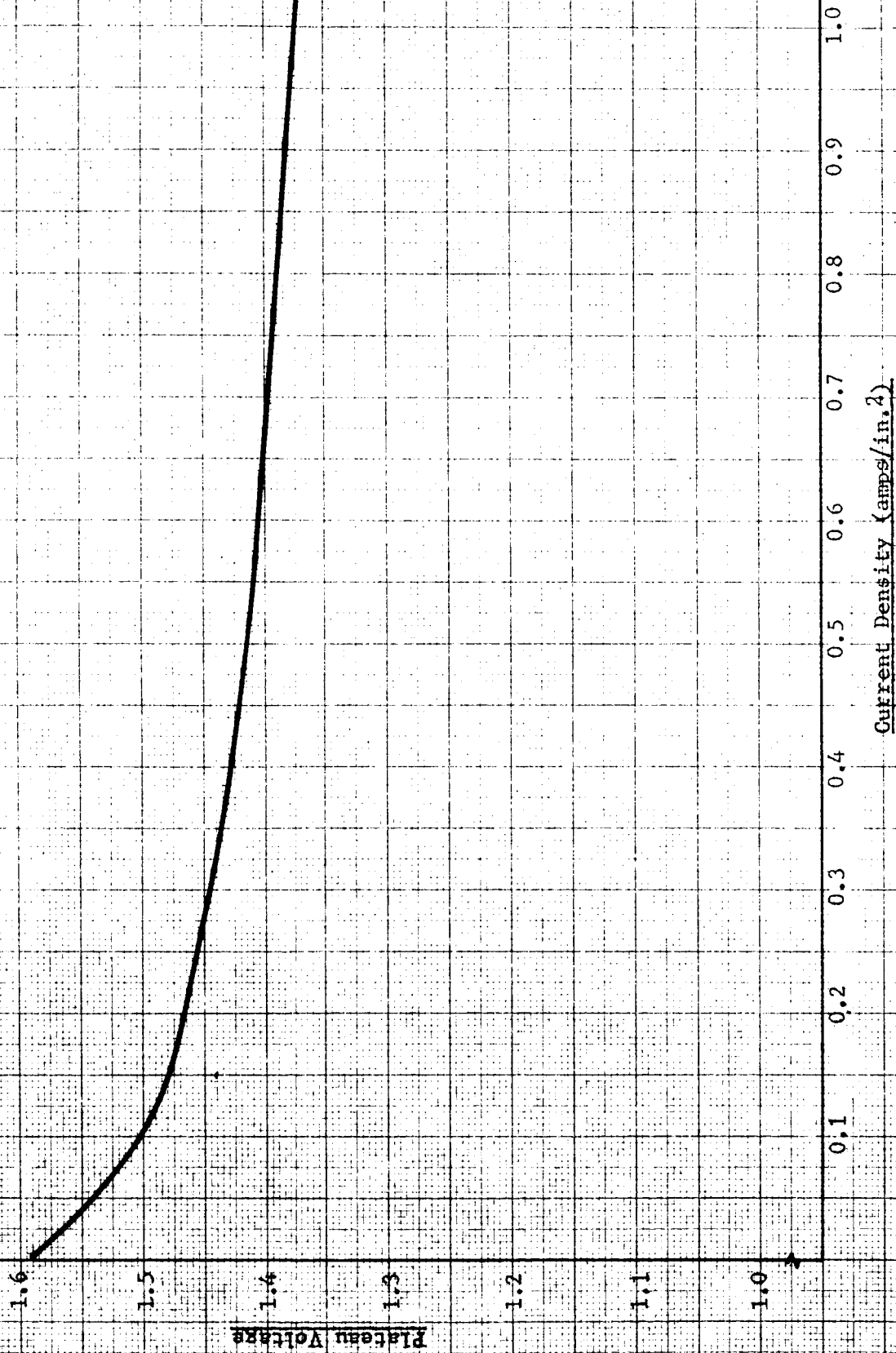
Other conditions affecting plateau voltage other than current density are essentially those which reflect cell internal impedance and are summarized below:

- 1) type of grid metal
- 2) method of plate-to-terminal connection
- 3) type of terminal
- 4) choice of separation
- 5) physical and electrical properties of the active materials
- 6) temperature
- 7) strength of electrolyte

Of the design conditions mentioned, current density, choice of grid material, and separators employed are probably most important, especially on high rate discharges.

FIGURE NO. 1

EFFECT OF CURRENT DENSITY
ON PLATEAU VOLTAGE



The following design factors, other than amount of active material, will affect cell capacity:

- 1) grid metal
- 2) type and number of layers of separator material
- 3) number of plates
- 4) active material density in grams per cubic inch
- 5) plate size after allowance for head room, etc.

Factors determining degree of voltage control are essentially those which affect cell internal resistance. The most important of these are as follows:

- 1) choice and number of wraps of separator material
- 2) active material density
- 3) method of active material formulation.

Energy density, in watt-hours per pound on other units, is affected by the following factors:

- 1) choice and number of wraps of separation
- 2) number of plates per cell
- 3) type of grid material
- 4) allowances for head room and tolerances
- 5) selection of hardware
- 6) size of battery unit.

Possible determining factors in length of useful activated stand are as follows:

- 1) type and method of separation
- 2) electrolyte concentration and quantity
- 3) electrolyte additives
- 4) active material additives and special treatments
- 5) temperature of storage.

It is obvious that several factors itemized above might affect either, favorably or adversely, more than one of the goals of the contract. As an example, the number of layers of membranes, or "closed type" separators, must increase with increasing activated stand. At the same time, these added layers of separation displace active material, thus decreasing cell capacity. Also, much greater cell internal electrical resistance may follow, resulting in poor voltage control as well as decreased plateau voltage.

In view of the many possible interrelations of design factors as exemplified above, it is anticipated that improvement in overall cell performance will result from optimization studies involving the several factors. This may be best accomplished by use of a statistically designed experiment. Factors which obviously are independent, that is, do not give rise to design "trade-offs", will be analyzed individually to facilitate calculations involved in the statistical approach.

B. Separator Evaluation

1. Properties of Ideal Separator

At the present state-of-the-art, the importance of separation material cannot be overestimated since it remains a limiting factor relative to the operation of the zinc-silver oxide system. For this reason, considerable effort has been expended on separation studies as indicated by the work schedule (see Appendix).

The ideal separation material would have the following characteristics:

- a. Low electrical resistance
- b. High absorbency
- c. Stability in concentrated potassium hydroxide
- d. Resistance to oxidation (caused by charged positive plate)
- e. High oxygen permeability (less important in the primary cells)
- f. Ability to retard migration of silver and zinc ions
- g. Good physical strengths
- h. Rapid wetting ability
- i. Stability of all properties over wide temperature ranges.

2. Separation Testing

Procedures have been outlined for quantitative determination of electrical resistance, absorbency, speed of wetting and the temperature dependence of these factors. Materials being considered for cell use will be tested in each of these areas.

a. Electrical Resistance

The apparatus used for determining areal resistance consists chiefly of a plastic reservoir filled with electrolyte and having parallel inert metallic electrodes at either of two opposing sides. This reservoir accepts a sample-holder which allows current to travel only through an opening of known area, 8.98 square inches. To determine resistance, one or more layers of various separators are fastened into the sample-holder, and direct current is passed through the apparatus at a measured rate. The voltage drop across the apparatus is measured. This voltage measurement is repeated as a blank, that is, with no separator material across the sample-holder opening. In this manner, slight effects of variation of temperature and slight changes in concentration may be eliminated. From the difference in the above voltage drops ($\Delta\Sigma$), the areal resistance, R, can be calculated according to the formula

$$R = \frac{(\Delta\Sigma) (8.98 \text{ in}^2)}{25 \text{ amperes}}$$

where the current through the apparatus is 25 amperes. This resistance is, of course, in units of ohm-inch². Figure No. 2 reveals a schematic diagram of this apparatus.

Data have been accumulated relative to single layers of separators of both open and closed types. These separators were tested for areal resistance at varying temperatures, speed of setting, and electrolyte absorbency. Table No. I is a list of these products and their outstanding identification features.

TABLE NO. I
SUMMARY OF COMMON SEPARATORS

Membranes	Wet Thickness	Description
133 Visking	.009 inch	Cellulosic
Fibrous Visking	.009 inch	Fibrous sausage casing
300 Cellophane	.003 inch	Regenerated cellulose
600 Cellophane	.0042 inch	Regenerated cellulose
PE Film 10/20	.002 inch	Irradiated polyethylene
PVA Film	.002 inch	Poly (vinyl alcohol)

Open Types	Wet Thickness	Description
Nylon	.003 inch	Woven 100% nylon
Nylon	.005 inch	Woven 100% nylon
Nylon	.011 inch	Woven 100% nylon
Pellon	.005 inch	Non-woven nylon
Pellon	.011 inch	Non-woven nylon
R-35-D	.00575 inch	Non-woven rayon
R-75-D	.0105 inch	Non-woven rayon

Phenomena not as yet fully explained have been noted in association with resistance measurements. It has been determined, for instance, that one layer of 300 Cellophane exhibits a resistance of 0.0053 ohm-in.² at +80° F, while two and three layers display resistances of 0.0180 and 0.0257 ohm-in.², respectively. In addition to this non-linear increase of resistance with increasing layers, a high apparent resistance has been noted for "open" separators. R-35-D, a non-woven absorbent, exhibits a resistance greater than that of single layers of PE Film, 300 Cellophane, or PVA Film, although it has a much greater gas permeability.

Exact quantitative permeability of separators to ionic species is not readily available, and it is possible that such data would explain the high resistance for some "open" separators. It is likely, however, that gas produced vigorously at the inert electrodes adheres to the surface of separators reducing the available current path. "Open" type separators provide a more favorable surface for this adherence of gas bubbles and could, therefore, display relatively higher resistance.

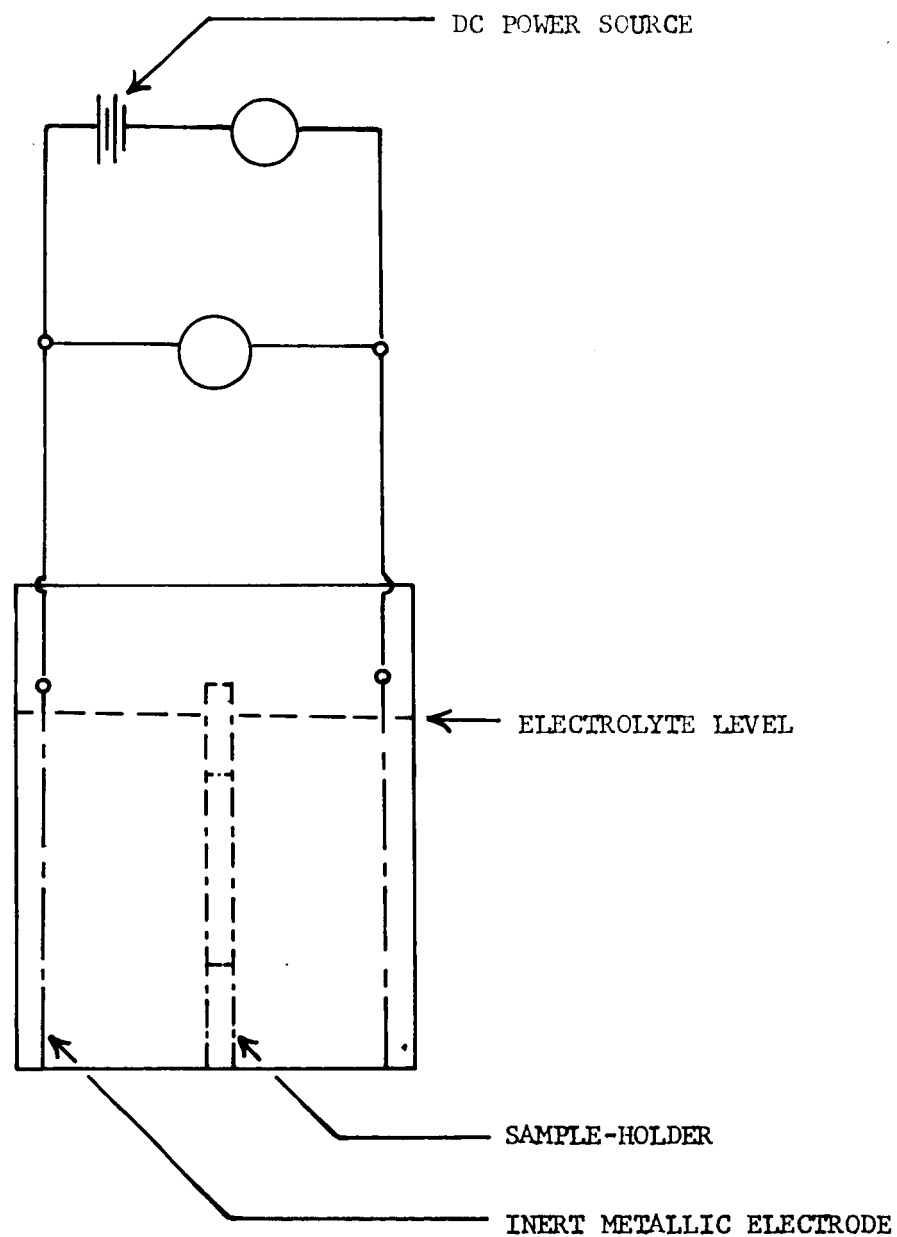


FIGURE NO. 2
RESISTANCE STUDY APPARATUS

Non-linearity in multiple layers could result from the gas adsorption, as described above, as well as air entrainment between layers, although every effort has been made to eliminate the latter effect. It is to be noted that high-rate discharges cause considerable gassing, so that resistance determinations as described above retain certain validity and must be considered a possibility in the operation of an electrochemical system using multi-layer insulation. However, in view of these facts, there are four alternatives for new resistance studies to be explored:

- 1) The resistance determination apparatus might be modified to deny accessibility of gases to the separation surfaces.
- 2) Electrodes of active, rather than inert, material could be used, resulting in eliminated or reduced gassing. This might present difficulties concerning polarization.
- 3) High frequency alternating current might be used, resulting in smaller current flow and hence only minor gassing.
- 4) Relative resistances might be determined by constructing three-plate test cells and determining impedance by the A.C. method. This would have the advantage of duplicating the deleterious environment encountered during activated cell life. Such test cells could be put on activated stand and the impedance determined at various intervals or states-of-charge.

Data accumulated relative to the resistance of multiple layers of separators are presented in Table No. II.

In view of the vastly increased electrical resistance of multiple layers of separation, it may be desirable to use single layers of low-resistance cellophane protected from the caustic electrolyte by application of another material.

Electrical resistance has been determined for single layers of 300 Cellophane protected on one and on both sides by application of polyvinyl alcohol. The resistances are 0.0071 and 0.0107 ohm-inch², respectively, compared to 0.0071 ohm-inch² for 0.002 inch PVA film, and 0.0054 ohm-inch² for 300 Cellophane alone. Further data relative to these membranes are contained in Table No. III, concerning life-cycle evaluation.

TABLE NO. II

RESISTANCE OF MEMBRANES
(Multiple layers at +80° F)

SEPARATOR	RESISTANCE (ohm-inch ²)		
	TWO LAYERS	THREE LAYERS	ONE LAYER
PE 40/20	.02255	.0628	.0056
300 Cellophane	.01796	.0257	.0054
.0015" PVA	.0287		.0072
600 Cellophane	.01796	.323	.0117
133 Visking	.0898	.531	.0226
Fibrous Visking			.0154
Polypor			.0826
.004" PVA			.0180
<u>Absorbents</u>			
R-35-D Viskon	.0055		.0084
Pellon	.0617		.0116
.004" Polypropylene			.0107
.009" Polypropylene	4.22		.0226
R-75-D Viskon	.0226		.0112
9526 Nylon	.0898		.00557
Webril	.0561		.0055
Dynel			.1123

TABLE NO. III

SEPARATOR LIFE CYCLE STUDY
(Cycles to Failure)

CELL NO.	NUMBER WRAPS	SEPARATOR COMBINATION	CYCLES TO FAILURE
1	1	9526 Nylon	879
	2	.002" Polyvinyl Alcohol film	
2	2	.002" Polyvinyl Alcohol film	347
3	1	9526 Nylon	347
	2	300 Cellophane	
4	1	9526 Nylon	347
	1	300 Cellophane	
	1	.002" Polyvinyl Alcohol film	
5	1 ea	9526 Nylon, 600 Cellophane	347
6	1 ea	Dynel, Fibrous Visking	347
7	1 ea	Dynel, 133 Visking	347
8	1	9526 Nylon	347
	1	300 Cellophane coated with PVA on one side	
9	1	9526 Nylon	171
	1	.002" Polyvinyl Alcohol film	
10	1	.002" Polyvinyl Alcohol film	144
11	1	9526 Nylon	749
	1	.004" Polyvinyl Alcohol film	
12	1	9526 Nylon	347
	3	PE 10/20 film	
13	1 ea	Dynel, Polypor, Polyvinyl Alcohol film	879
14	1	9526 Nylon	1232
	3	PE 10/20 film	
15	1	9526 Nylon	347
	2	PE 10/20 film	
16	1 ea	9526 Nylon, 300 Cellophane and Polyvinyl Alcohol film	1232
17	1 ea	PE film (special), 9526 Nylon	528
18	2	PE film (special)	528
	1	9526 Nylon	

b. Cycle Testing of Separation

The use of cadmium rather than zinc negative material eliminates the likelihood of cycle failure because of loss of active material. This insures that the number of cycles to failure of an individual cell indicates the relative ability of the particular separator combination to withstand the deleterious environmental conditions encountered. The charge portion of each cycle adds a small overcharge to insure ability of separators to resist silver ionic migration at high oxidation potentials.

While the number of cycles to failure is of no specific value in itself, comparison of cycle life of various test cells reveals the relative quality of individual semi-permeable membranes and separator combinations, and aids in selecting the optimum for use in the primary cell. The test serves to accelerate evaluation of separator materials under actual operating conditions whereby the most promising types can be selected for study in regular cell assemblies.

Table No. I lists the combinations of separators used. Periodic end-of-charge and end-of-discharge voltages were recorded. At fixed intervals, the test cells were allowed to stand forty-eight hours in the charged condition for the purpose of monitoring open circuit voltages. The criterion for failure is reversal upon discharge or an open circuit voltage lower than that associated with the monovalent silver oxide - cadmium couple following the stand period.

These separation evaluation studies were designed primarily to evaluate the ability of various insulation materials to retard the penetration of silver to form a metallic short circuit path. Since zinc plates cannot be used because of their short life, these tests do not disclose the ability of separator materials to withstand penetration by zinc which, in the silver-zinc system, is also a major mode of failure. However, it is assumed that a separator material best capable of retarding silver penetration will likewise be successful in alleviating effects of zinc penetration.

All test cells employed in the rapid-cycle separator evaluation study have failed. A summary of cycle data is revealed by Table No. III.

"Post mortem" inspection revealed that considerable sloughing of active material had occurred at the negative plates, and it cannot be stated with absolute certainty that cell failure in every case resulted from separator failure. Results confirm the obvious assumption that cell life increases with the number of layers of separation. It is also revealed that multiple layers of separation are capable of withstanding the deleterious effects of caustic environment and oxidation potentials for periods of months at room temperatures. The data do not reveal any obvious method of selecting the optimum combination of separator materials for operation over a range of temperatures, a factor that must be considered since variation in coefficient of expansion of separator material can be expected to alter subsequent performance characteristics.

c. Speed of Wetting

In the procedure used for determination of separator resistance, readings are taken at time intervals until no further change in resistance is noted. This allows observation of the speed of wetting of the various materials. As there is no common criterion for this property, data are interpolated to reveal the soak time after which a separator displays a resistance (R_2) equal to twice its final stable resistance. These data are presented in Table No. IV. It is to be noted that these wetting speeds are only realized under ideal conditions of electrolyte accessibility. Wetting speed in a cell can vary greatly with actual cell design.

d. Electrolyte Absorption and Retention

Another important quality of separators is electrolyte retention. To evaluate this characteristic, samples of various separators were dried, weighed accurately, soaked for seventy-two hours in 1.300 specific gravity potassium hydroxide, removed, and reweighed. Before the second weighing, the sample was held vertically a few seconds until the excess KOH formed droplets at the edge of the sample. Only these droplets were blotted to avoid removing electrolyte held in the pores of the sample. A summary of absorption data is presented in Table No. V.

Another importance of electrolyte absorption lies in the determination of optimum amount of electrolyte to be included in a cell. It is anticipated that the optimum quantity of electrolyte for this system is that which saturates both the plates and separator. However, cell tightness will affect the absorption properties of separator materials.

TABLE NO. IV

EFFECT OF TEMPERATURE
ON SEPARATION PROPERTIES

SEPARATOR MATERIAL	RESISTANCE (ohm-inch ²)			TR ₂ (minutes)**		
	0° F	+80° F	+120° F	0° F	+80° F	+120° F
133 Visking	0.054	0.0225	0.011	30	4	4
Fibrous Visking		0.0143			2	
300 Cellophane	0.0269	0.0053	*	1.75	0.5	+
600 Cellophane	0.025	0.0116	0.00718	4	0.5	0.5
PE Film-307	0.0898	0.0071	*	2	1	+
PVA Film	0.0322	0.0071	0.0071	20	3	1.2
0.005" Nylon		0.0024			+	
0.011" Nylon		0.0055			+	
0.005" Pellon		0.0071	0.0071		+	1 min.
0.012" Pellon	0.0269	0.0116	0.00359	0.55	+	1 min.
R-35-D	0.00898	0.0084	*	1	+	0.85
R-75-D	0.00898	0.0112	0.00718	2	1	+

* Resistance too small to be detected.

+ Resistance reached final almost immediately.

Blank spaces indicate data not yet obtained.

** TR₂ represents the soak time required for the sample to attain a resistance equal to twice its final stable value.

TABLE NO. V

CHARACTERISTICS OF VARIOUS SEPARATOR MATERIALS
(one layer at +30° F - 72-hour soak)

SEPARATOR MATERIAL	DIMENSIONS (in.)	AREA (square inches)	DRY WEIGHT (grams)	WET WEIGHT (grams)	ELECTROLYTE RETENTION BASIS-1 in ²		ABSORBENCY Wt. Wet - Wt. Dry Wt. Dry	THICKNESS, WET (inches)	RESISTANCE	
					Gm. KOH	cc KOH			ohm-in ²	ohm-in*
PE Film-307	12.0 x 12.0	144	3.1138	11.0750	0.0552	0.0423	2.56	0.002	0.0071	3.05
PVA Film	6.25 x 12.0	75	2.5428	7.4120	0.00649	0.0499	1.84	0.002	0.0071	3.05
300 Cellophane	6.0 x 12.0	72	1.6628	7.6100	0.0826	0.0635	3.58	0.003	0.0053	1.76
600 Cellophane	11.0 x 11.0	121	4.2462	18.0970	0.1140	0.0830	3.26	0.0042	0.0116	2.52
Visking 133	5.0 x 6.0	30	2.1830	7.4130	0.1743	0.1340	1.94	0.009	0.0225	2.52
Fibrous Visking	5.0 x 6.0	30	1.7290	6.1970	0.1489	0.1145	2.52	0.009	0.0143	1.59
R-35-D	6.0 x 6.0	36	0.6517	7.0190	0.1768	0.1360	7.96	0.00575	0.0034	1.46
R-75-D	2.5 x 6.5	16.25	0.6120	5.7050	0.3134	0.2410	8.33	0.0105	0.0112	1.07
0.003" Nylon	2.5 x 7.0	17.5	0.2580	1.0384	0.0445	0.0342	3.03	0.003	0.0014	0.464
0.005" Nylon	12.0 x 12.0	144	4.3434	10.4103	0.0421	0.0323	1.400	0.005	0.0024	0.480
0.011" Nylon	2.63 x 6.5	16.01	0.6561	1.5260	0.0541	0.0416	1.305	0.011	0.0055	0.500
0.005" Pellon	10.0 x 10.0	100	3.6164	10.2160	0.0659	0.0507	1.83	0.005	0.0071	1.420
0.011" Pellon	6.0 x 6.0	36	1.3800	12.8850	0.3195	0.2458	8.33	0.011	0.0116	1.055
Polypor	9.0 x 9.0	81	2.9514	10.3075	0.0903	0.0698	1.83	0.00425	0.00826	1.94

* This value is obtained by dividing the resistance in the preceding column (ohm-inch²) by the wet thickness in inches.

C. Evaluation of Capabilities of Present Design

1. Cell Variables and Cell Testing

In order to evaluate the current state-of-the-art and establish a point of reference, a series of cells was constructed similar to those employed in the Eagle-Picher MAP-4101 battery, which yields 10 to 12 ampere-hours at the 15-minute rate. Four combinations of separators were employed as follows:

Group I, Cells 11-15

One wrap Polypor next to the positive plate
Two wraps 300 Cellophane over the Polypor
One wrap R-35-D absorbent on the negative

Group II, Cells 21-25

Three wraps PE Film on the positive
One wrap R-35-D on the negative

Group III, Cells 31-35

One wrap 9526 Nylon on the positive
Two wraps 300 Cellophane over the Nylon
One wrap R-35-D on the negative

Group IV, Cells 41-45

One wrap 133 Visking on the positive
One wrap R-75-D on the negative

These combinations were chosen since they allow comparison of three combinations of separators employed in Groups I, II, and III to that of the standard, Group IV. Approximately the same total wet separator thickness exists in each case, allowing identical active material weights and theoretical capacities. Five cells of each type were constructed, using standard production line techniques to insure reproducibility. All cells were activated on August 19, 1963 with 20 cc of 1.400 specific gravity potassium hydroxide. One cell of each type was discharged at 37 amperes immediately following a two-hour soak period. (This rate represents 0.815 ampere per square inch, or the 15 to 20-minute rate.)

A tentative goal was to obtain the maximum possible capacity with a plateau voltage of 1.40 volt per cell at the 20-minute discharge rate, following a minimum of thirty days activated stand at room temperature. It is also desirable to develop discharge characteristics such that it is unnecessary to pre-load cells in order to obtain voltage control and avoid the so-called "inverted voltage-spike" associated with high-rate discharges.

This series of discharges has been completed. Activated stand times of two hours, two weeks, three weeks, one month, and eight weeks were included. Results of these discharges are illustrated by Figure Nos. 3, 4, 5 and 6.

All cells displayed a significant decrease in capacity after one month activated stand. The final discharge, after eight weeks activated stand, revealed that only Series IV cells employing 133 Visking next to the positive plate, exhibited a significant decrease in capacity between one and two months activated stand. The remaining cells, in fact, displayed higher voltage and greater capacity relative to one month activated stand. Data relative to capacity as a function of activated stand are displayed by Figure No. 7. Smooth curves were not employed in presenting these data because present knowledge is not sufficient to formulate an explicit function relating to capacity and activated stand. The apparent maximum in the three week stand area may have resulted from a slightly different heat sink arrangement, the effect of which is magnified at the reasonably high discharge rate which was employed.

2. Conclusions

The following conclusions can be drawn from the results of this series of discharges.

a. PE Film

Three layers of the PE Film (an ion exchange membrane) proved to be undesirable for this specific application. Figure No. 4 reveals cells employing three layers of PE Film in addition to 9526 Nylon on the positive plates displayed in general low voltage and poor voltage control. "Post mortem" of PE cells revealed that all layers of the film contained silver. Two layers apparently would not have been sufficient to prevent silver from reaching the zinc plate.

b. Polypor and 300 Cellophane

Use of one layer of Polypor, another ion-exchange membrane, along with two layers of 300 Cellophane, revealed no outstanding advantages. The Polypor membrane did not prevent silver migration to any appreciable extent. The cellulosic membrane showed considerable silver loading and physical deterioration.

c. Woven Nylon and 300 Cellophane

The combination of 9526 Nylon with three wraps of 300 Cellophane appeared to be the most satisfactory one. Both voltage and capacity were very good at the two and three week stand levels. Discharge voltage was also "flat" indicating good voltage control. Voltage and capacity for the longer stand periods were comparable to that yielded with other separator combinations.

FIGURE NO. 3
 PRIMARY ZINC - SILVER OXIDE STAND CHARACTERISTICS
 DISCHARGED AT 0.8 AMPERE PER SQUARE INCH

1 wrap Polypor
 2 wraps 300 Cellophane
 1 wrap R-35-D Viskon

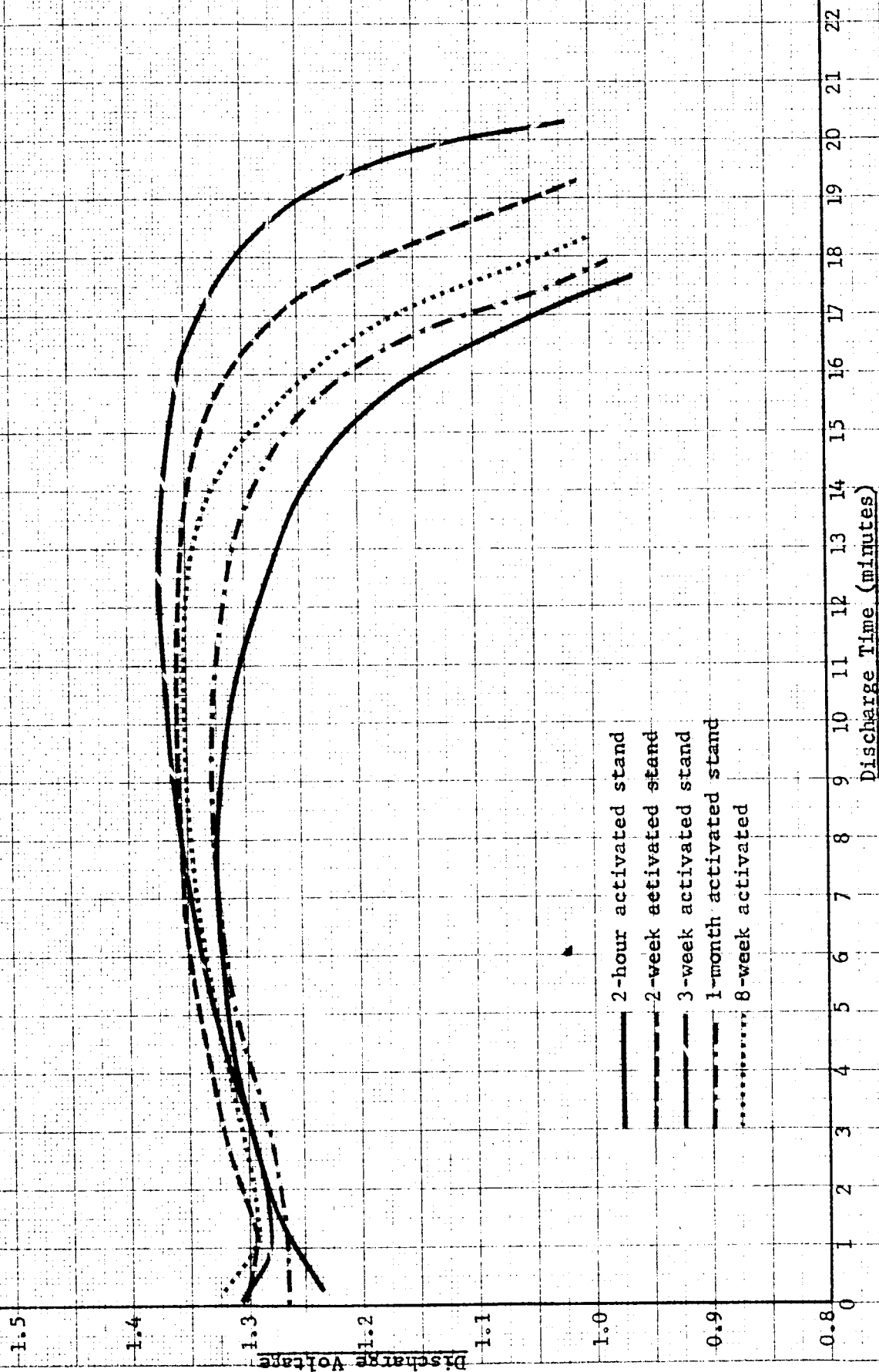


FIGURE NO. 4
PRIMARY ZINC - SILVER OXIDE STAND CHARACTERISTICS
DISCHARGED AT 0.8 AMPERE PER SQUARE INCH
3 wraps PE film
1 wrap R-35-D Viskon

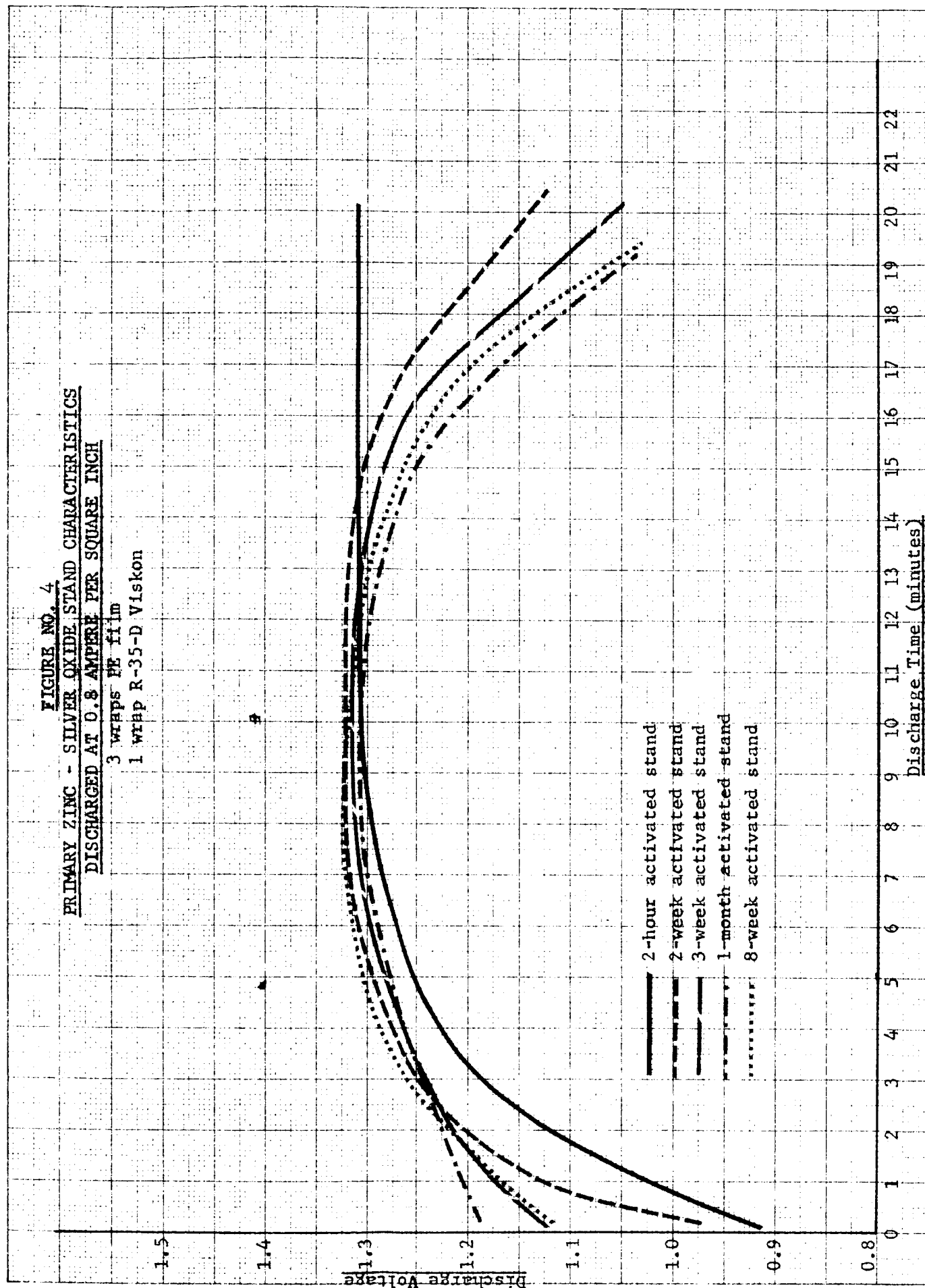


FIGURE NO. 5
PRIMARY ZINC - SILVER OXIDE STAND CHARACTERISTICS
DISCHARGED AT 0.8 AMPERE PER SQUARE INCH
1 wrap Nylon
2 wraps 300 Cellophane
1 wrap R-35-D Viskon

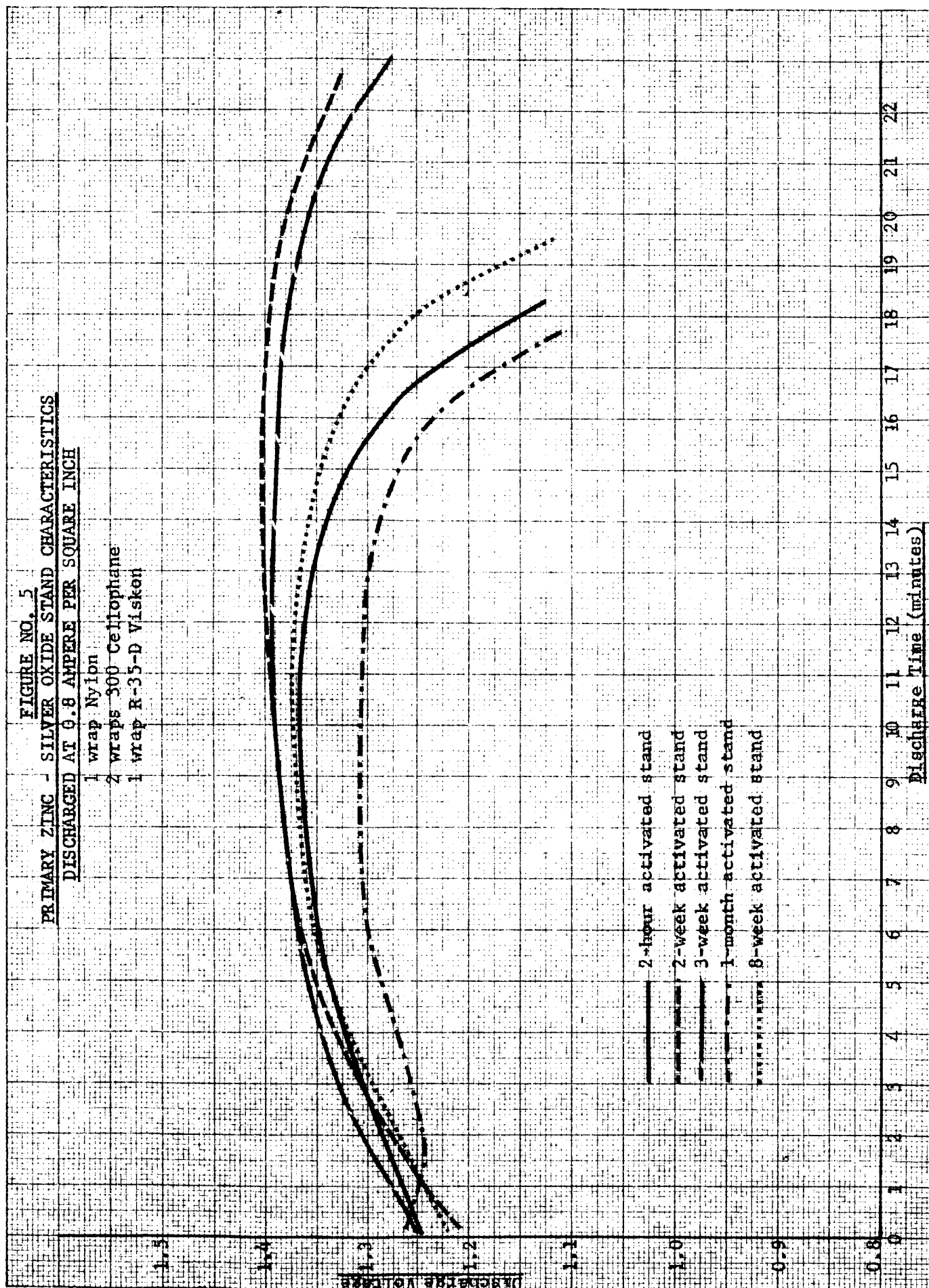


FIGURE NO. 6

PRIMARY ZINC - SILVER OXIDE STAND CHARACTERISTICS

DISCHARGED AT 0.8 AMPERE PER SQUARE INCH

1 wrap 133 Visking

1 wrap R-75-D Viskon

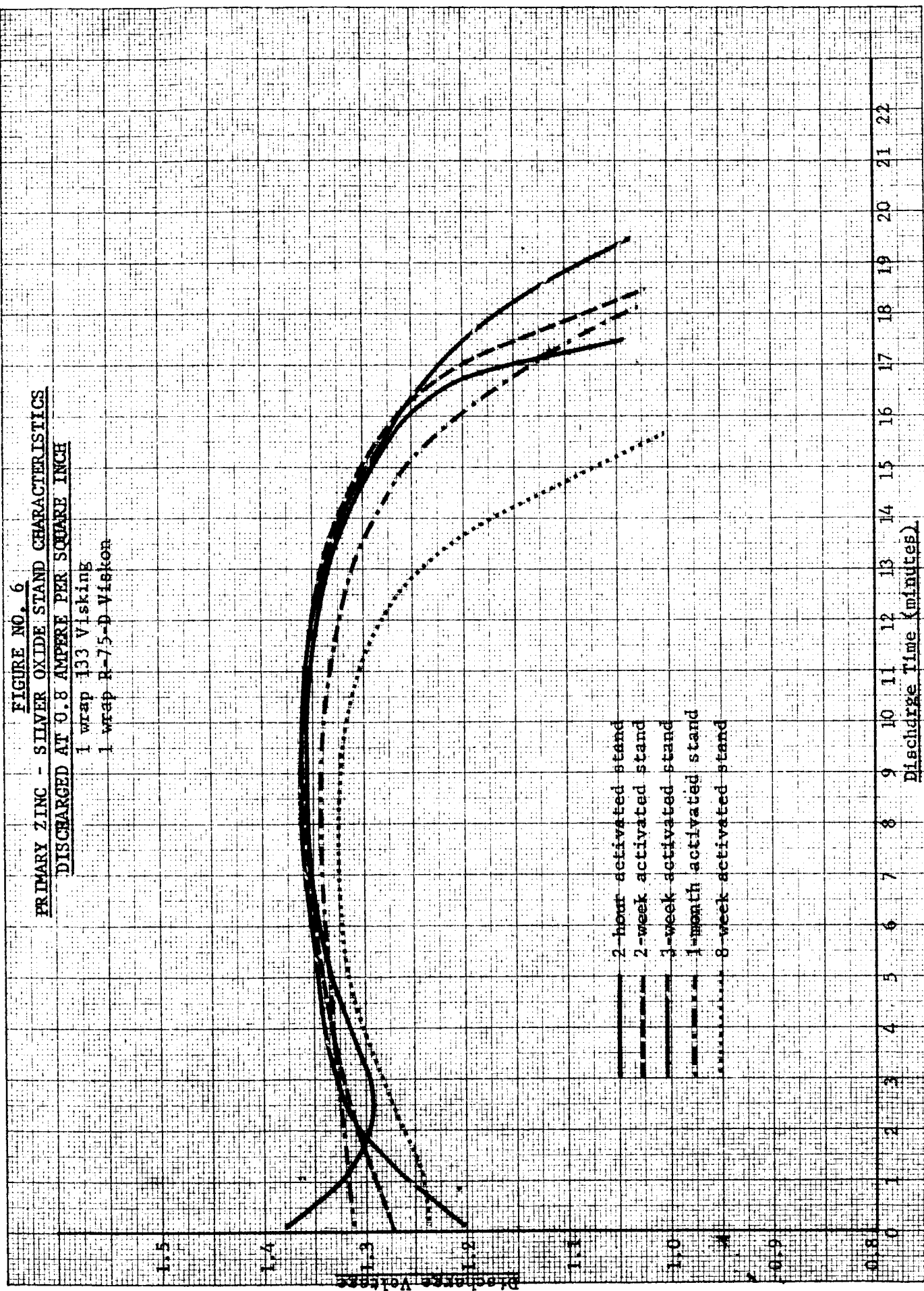
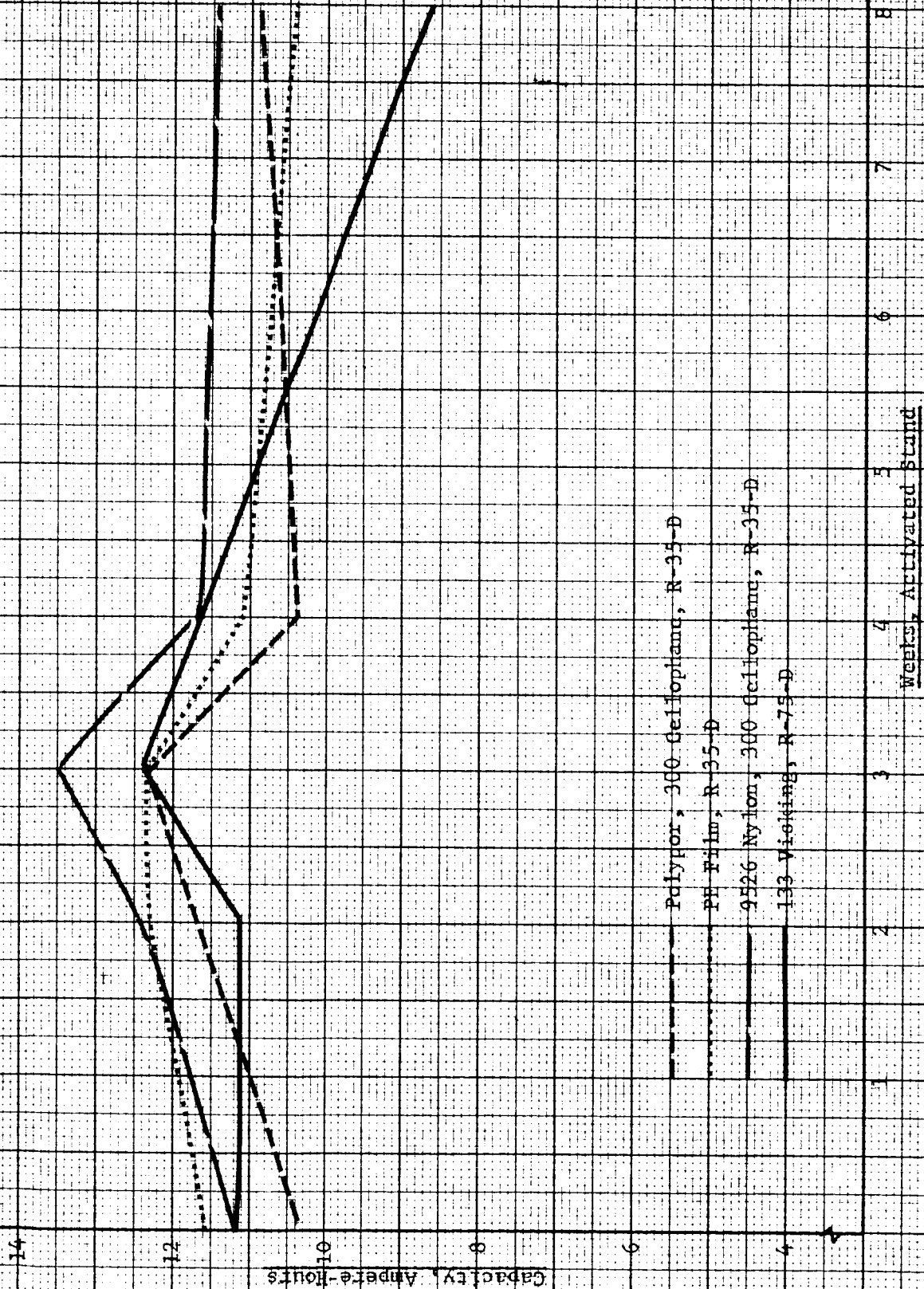


FIGURE NO. 7
EFFECT OF STAND ON CELL CAPACITY
DISCHARGED AT 0.5 AMPERE PER SQUARE INCH



d. Control Cell

With the exception of the final discharge after two months stand, the least variation of voltage and capacity with time was exhibited by the "control" cell. This cell employed one layer of 133 Visking next to the positives. However, voltage and capacity were low and "flat" discharge voltage was not obtained.

In addition, silver crystals which formed next to the positive plates locally forced the Visking from the plates, causing uneven plate discharge and damage to the Visking. Partial shorting resulted after eight weeks stand.

This is in agreement with other data which indicate the desirability of employing an "open" material such as nylon, woven or non-woven, between the positive plate and the membranous separator. This combination not only prevents damage to the membrane by crystalline growth, but also encourages rapid activation and uniform plate discharge.

Some caution must be used in the interpretation of the above data. The fact that no complete cell failures were displayed in the limited test samples does not provide a basis for the statistical certainty of eight or even four-week activated stands. It is indicated, however, that retention-of-charge characteristics as presented may be achieved under laboratory conditions.

This series of cells was constructed to indicate performance and stand characteristics of the "control" cell and cells employing three other separator combinations, as described. It was anticipated that such a series of discharges would indicate intervals of activated stand at which discharges could be conducted to produce the maximum amount of useful data. It was not intended to establish the reliability of the cell, although reliability determination is planned for the final cell design.

It has been pointed out that activation of individual cells at room temperature does not produce as great a temperature increase (which results from chemical reactions) as would occur in a complete battery. This explains the fact that previous data do not indicate reliable battery performance following stands as great as were obtained on the test cells of this series.

As the stand test was not carried to cell failure, the cell cumulative failure distribution could not be determined.

The fact that several individual cells might be expected to stand satisfactorily for periods of two months precludes the total use of room temperature for successive tests as might be required by a program of optimization.

There are three major causes of cell failure in the silver oxide - zinc system which are as follows:

1. loss of oxygen from the charged positive plate;
2. loss of negative capacity through dissolution of zinc or other mechanisms;
3. internal shorting because of silver and/or zinc migration, separator breakdown, etc.

It is fundamental that failures resulting from these causes are accelerated by elevated temperatures. Discharge characteristics such as voltage magnitude and voltage control are adversely affected by low temperatures. It is desirable, then, to obtain a cell design which will allow satisfactory activated stand at high temperatures and satisfactory discharge characteristics at lower temperatures. The cell which serves best over a range of temperatures will undoubtedly represent a compromise between these two conditions.

It has been decided that in order to speed accumulation of data, a stand temperature of +130° F shall be used. The cell which survives this environment the longest period of time also would be expected to survive longer at lower temperatures.

It is anticipated that failures will occur comparatively rapidly at +130° F. This will allow a rapid determination of the cell best suited for long stand times.

If, in addition, sets of cells are removed from +130° F stand at varying time intervals, stabilized at +70° F and discharged, the discharge characteristics can be determined as a function of stand time. It is likely that sufficient data may be accumulated to correlate stand time, stand temperature and cell capacity such that capacity following +70° F stands may be closely estimated from capacity following stand at +130° F. This will allow estimation of the time at which cell capacity will fall below any selected value.

D. Fractional Factorial Experiment

The evaluation of the many apparent variables by the "classical" methods may involve evaluation tests on great numbers of experimental cells. Even so, interactions (the measure of the extent to which factors fail to act independently) cannot be evaluated or even detected by varying only one factor at a time. This makes desirable the use of a "factorial" or "fractional factorial" experiment. The factorial experiment is based upon the principle of varying more than one factor simultaneously and in a predetermined manner.

Factors considered for evaluation and the levels of each factor are listed by Table No. VI. One factor includes two levels: Factor A has four levels, and the remaining factors each have three levels. A full factorial experiment would require $4 \times 2 \times 3^6$ test cells, if all interactions

TABLE NO. VI

TEST VARIABLES AND THEIR LEVELS

A. Additives to Electrolyte

- A₀ - None
- A₁ - 1% gel
- A₂ - MnO₂ (at saturation)
- A₃ - LiOH (at saturation)

B. Electrolyte Concentration

- B₀ - 35%
- B₁ - 40%
- B₂ - 45%

C. Positive Material Density (gms./cu.in.)

- C₀ - 68
- C₁ - 74
- C₂ - 80

D. Positive Grid Metal

- D₀ - 4/0 Ni
- D₁ - 4/0 Ag

E. Negative Material Density

- E₀ - 40
- E₁ - 45
- E₂ - 50

F. Additive Content in Negative Plate *

- F₀ - 1%
- F₁ - 2%
- F₂ - 4%

G. Negative Grid Metal

- G₀ - Copper (4/0)
- G₁ - Silver Flashed Cu (4/0)
- G₂ - Silver (4/0)

H. Negative Formulation **

- H₀ - Pasted
- H₁ - Sponge
- H₂ - Metallic

* The nature of this additive and its introduction into the negative plate is proprietary.

** These are descriptive names referring to three types of negative plates prepared by proprietary procedures.

were to be quantitatively determined. Such a large number of cells is beyond the scope of this project. Therefore, a fractional factorial pattern was chosen, involving twenty-seven distinctly different cells. This pattern is displayed by Table No. VII.

TABLE NO. VII
FRACTIONAL FACTORIAL PATTERN
TWENTY-SEVEN CELL GROUP

CELL NO.	L E V E L S							
	A	B	C	D	E	F	G	H
1	0	0	0	0	0	0	0	0
2	0	1	1	1	2	1	2	2
3	0	2	2	0	1	2	1	1
4	1	0	0	0	0	1	1	1
5	1	1	1	0	2	2	0	0
6	1	2	2	1	1	0	2	2
7	2	0	0	1	0	2	2	2
8	2	1	1	0	2	0	1	1
9	2	2	2	0	1	1	0	0
10	1	0	1	1	1	0	0	1
11	1	1	2	0	0	1	2	0
12	1	2	0	0	2	2	1	2
13	3	0	1	0	1	1	1	2
14	3	1	2	1	0	2	0	1
15	3	2	0	0	2	0	2	0
16	3	0	1	0	1	2	2	0
17	3	1	2	0	0	0	1	2
18	3	2	0	1	2	1	0	1
19	2	0	2	0	2	0	0	2
20	2	1	0	0	1	1	2	1
21	2	2	1	1	0	2	1	0
22	2	0	2	1	2	1	1	0
23	2	1	0	0	1	2	0	2
24	2	2	1	0	0	0	2	1
25	0	0	2	0	2	2	2	1
26	0	1	0	1	1	0	1	0
27	0	2	1	0	0	1	0	2

This will not allow quantitative determination of all interaction or "trade-offs", but will allow evaluation of individual factors, and at the same time, may indicate the presence of certain desirable or undesirable combinations. Factors or combinations of factors that are revealed to be significant may then be isolated and studied further with smaller increments of variance to accurately locate optimum combinations of conditions.

1. Discussion of Variables

The following is a summary of the methods in which test variables might be expected to influence characteristics of the primary zinc - silver oxide system.

a. Additives to the Electrolyte

Lithium hydroxide may retard carbonation of the electrolyte and active material. Its effect is expected to be greater at elevated temperatures.

Manganese dioxide, sparingly soluble in caustic solutions, may retard hydrogen liberation at the negative plate.

The gelatinous electrolyte additive is expected to retard bulk movement of excess electrolyte and discourage relocation of zinc throughout the cell.

b. Electrolyte Concentration

Characteristics of the KOH-H₂O system as a function of "strength" and temperature are exhibited in Figure Nos. 8 and 9. It can be seen that resistivity of a KOH-H₂O solution is at a minimum in the region of 1.300 specific gravity or 31% by weight of KOH in water.

TABLE NO. VIII

CELL CLASSIFICATION WITH RESPECT
TO ELECTROLYTE CHARACTERISTICS

	B ₀	B ₁	B ₂
A ₀	1, 25	2, 26	3, 27
A ₁	4, 10	5, 11	6, 12
A ₂	7, 19, 22	8, 20, 23	9, 21, 24
A ₃	13, 16	14, 17	15, 18

"A" represents electrolyte additives

"B" represents electrolyte concentration

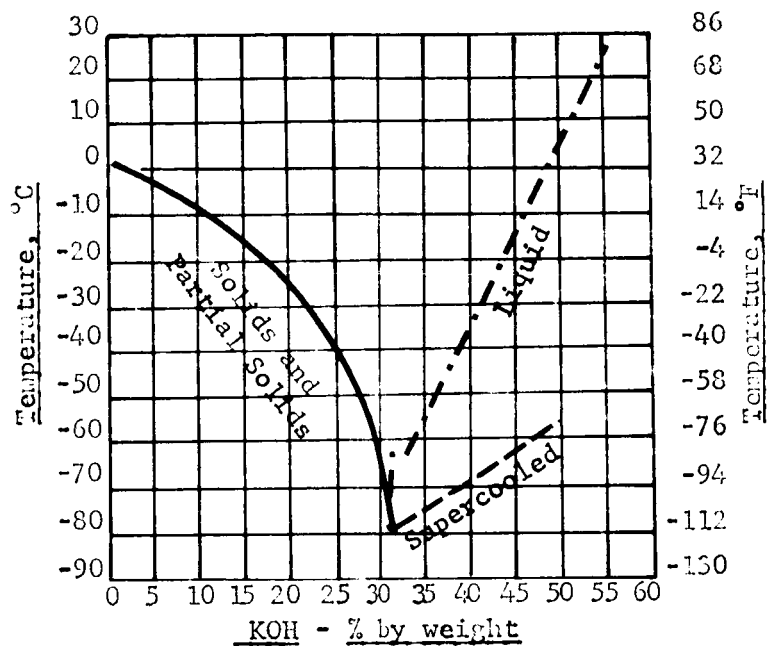


FIGURE NO. 8
KOH-H₂O PHASE DIAGRAM

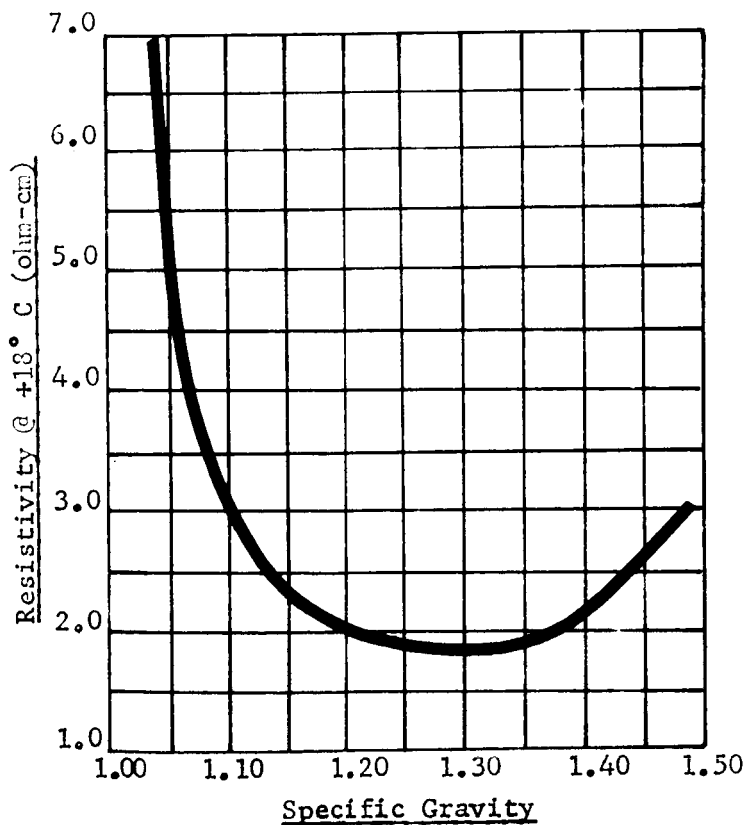


FIGURE NO. 9
RESISTIVITY OF
KOH-H₂O SOLUTION

A cell employing a 31% solution of KOH as electrolyte would be expected to display the highest voltage at any temperature or current density of discharge. The freezing point of the caustic electrolyte is also at a minimum for this strength as is indicated by the phase diagram, Figure No. 3. Solubility of the silver species is also greatest at this concentration; therefore, higher concentrations are commonly employed in cells intended for long shelf life or high temperature stands.

c. Positive Material Density

Thickness, to which the positive material is compressed, affects the mechanical bonding between particles as well as the effective surface area of the resulting silver oxide. These changes are reflected in the availability of the potential electrochemical energy and, hence, the capacity efficiency of the positive active material.

d. Positive Grid Material

Choice of grid metal determines the electrical resistance of the grid. This is reflected in the discharge voltage.

e. Negative Material Density

Apparent density is important in the zinc negative for the same reasons as are associated with the positive. Efficiency of the negative active material has been observed to decrease considerably at apparent densities in excess of 50 grams per cubic inch (probably depending somewhat upon particle size).

f. Additive Content in Negative Plate

The effect of this proprietary additive is to retard hydrogen evolution on stand, thus decreasing the dissolution of zinc and thereby increasing activated stand. This is done at some slight sacrifice of discharge voltage.

g. Negative Grid Metal

As with positive grid metal, choice of negative grid material is reflected in the discharge voltage. Hydrogen overvoltage may also be considered in choosing grid metal. For example, there is a tendency for unprotected copper to corrode on stand, resulting in higher internal resistance and copper contamination.

h. Negative Formulation

There is little previous data relating to the method of negative material formulation to long stand application. It now appears to be advantageous to evaluate zinc produced by a fourth proprietary procedure, recently put into use at Eagle-Picher.

2. Cell Construction

a. Properties of Grid Metal

Of particular importance in any element design is the choice of grid and separator materials. Grid metal displaces active material; therefore, it is desirable to establish the minimum amount of grid metal required for certain current-carrying capabilities.

The following is a discussion of the relative current-carrying capacities of various commonly used grid materials. An elementary formula for the resistance of a conductor is

$$R = \frac{L}{A} \quad \text{where}$$

P, L, and A are the resistivity, length, and area, respectively, of the conducting material. We shall assume that a current flows unidirectionally in the grid, in the direction of the terminal. Taking a unit length of one inch in the direction of current travel, the resistance may easily be determined for a square inch of grid metal. Table No. IX contains data relative to these determinations.

TABLE NO. IX

RESISTANCES OF COMMON METALLIC GRIDS

GRID METAL	RESISTIVITY	In ³ METAL PER SQ. IN. OF GRID	OHMIC RESISTANCE OF 1 in ²	RESISTANCE RELATIVE TO 4/0 Ag R'
4/0 Ag	1.62 x 10 ⁻⁶ ohm-cm	0.00244	1.69 x 10 ⁻³	1.00
3/0 Ag	1.62 x 10 ⁻⁶ ohm-cm	0.00192	2.14 x 10 ⁻³	1.27
2/0 Ag	1.62 x 10 ⁻⁶ ohm-cm	0.00157	2.62 x 10 ⁻³	1.55
1/0 Ag	1.62 x 10 ⁻⁶ ohm-cm	0.00069	5.96 x 10 ⁻³	3.53
4/0 Cu	1.72 x 10 ⁻⁶ ohm-cm	0.00295	1.48 x 10 ⁻³	0.876
4/0 Ni	7.24 x 10 ⁻⁶ ohm-cm	0.00171	1.03 x 10 ⁻²	6.33
4/0 Zn	5.92 x 10 ⁻⁶ ohm-cm	0.00371	4.06 x 10 ⁻³	2.40

These data are not absolutely correct because of the cross-sectional area of the component wire variance with the type grid. It is readily apparent, however, that 4/0 nickel grid displaces more active material than 2/0 silver, but has a resistance more than four times that of 2/0 silver grid and 6.33 times that of 4/0 silver. Although copper is slightly more resistive than silver, one square inch of 4/0 copper, for example, has a

resistance of 0.88 times that of an equal area of 4/0 silver grid. This is because the copper grid contains a greater volume of conductive metal per unit area. A cell employing 4/0 silver grid in the positive and 4/0 copper in the negatives would be expected to exhibit the greatest discharge voltage.

Degree of over-pasting also affects conductivity. The same grams per square inch on 4/0 require more over-pasting than lighter grids. It is also to be noted that expanded grid materials vary considerably between manufactures.

b. Miscellaneous Construction Variables

Figure Nos. 10 and 11 correlate void space and apparent density in grams per cubic inch for the zinc and divalent silver oxide plates. Apparent density affects the efficiency of the active material as well as the quantity of electrolyte required to wet the electrode. Such data can be used in conjunction with similar data for separator materials to estimate the electrolyte requirement for a cell.

Figure No. 12 relates physical characteristics of the zinc plate for two commonly used negative grid materials, silver and copper. These data were used to determine the thickness to which zinc plates were compressed so that they complied to the apparent density required for the design experiment.

c. Special Process Zinc

A limited supply of metallic zinc, prepared by a proprietary procedure which shall be referred to as E.P. Process No. 4, was used in test cells during the sixth contract month to determine whether it merited more extensive testing. Zinc prepared by this process is characterized by large particle size and great adhesion to the grid metal.

The response of the discharge voltage to varying discharge current densities was of particular interest.

Figure No. 13 reveals the effect of current density on discharge voltage for "control" cells and those using zinc prepared by E.P. Process No. 4. Data employed in this figure were obtained by discharging cells to the voltage associated with monovalent silver oxide, then surging the cells at various discharge currents. Surges were of short duration to prevent internal heating. It can be seen that the control cells exhibited higher voltages. This can be explained in part by the greater particle size and, therefore, smaller effective surface area of the special zinc being tested.

The results indicate that redesign of the cell to produce more surface area would be necessary before this zinc could be used satisfactorily at the 15 to 30-minute discharge rate. This would, in turn, reduce cell capacity undesirably. Also, it disclosed an approach for improving voltage of the standard design through adjustment of the apparent density of spongy zinc.

FIGURE NO. 10
VOID SPACE IN THE ZINC PLATE
AS AFFECTED BY APPARENT DENSITY

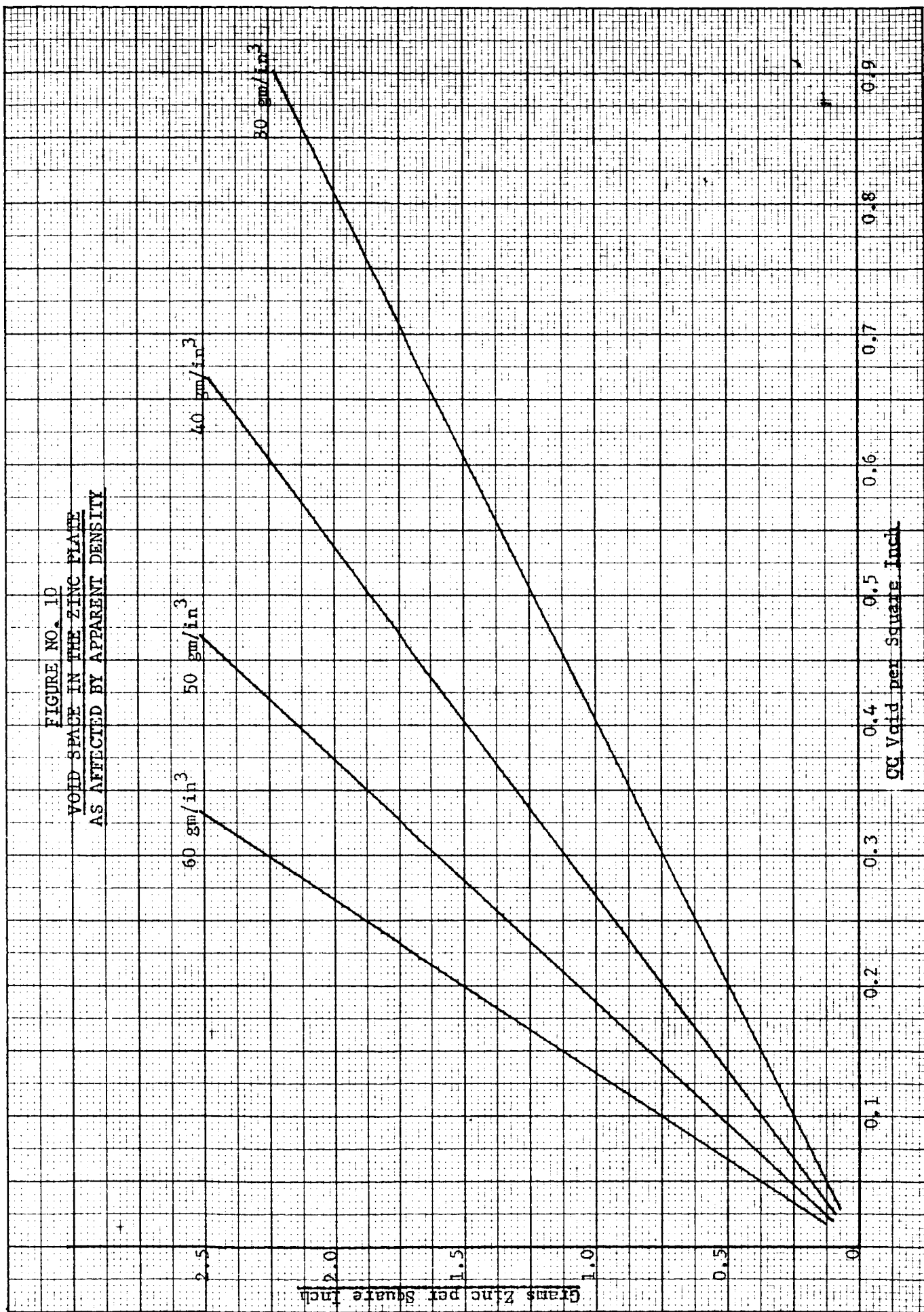


FIGURE NO. 11
VOID SPACE IN THE DIVALENT SILVER OXIDE PLATE
AS AFFECTED BY APPARENT DENSITY

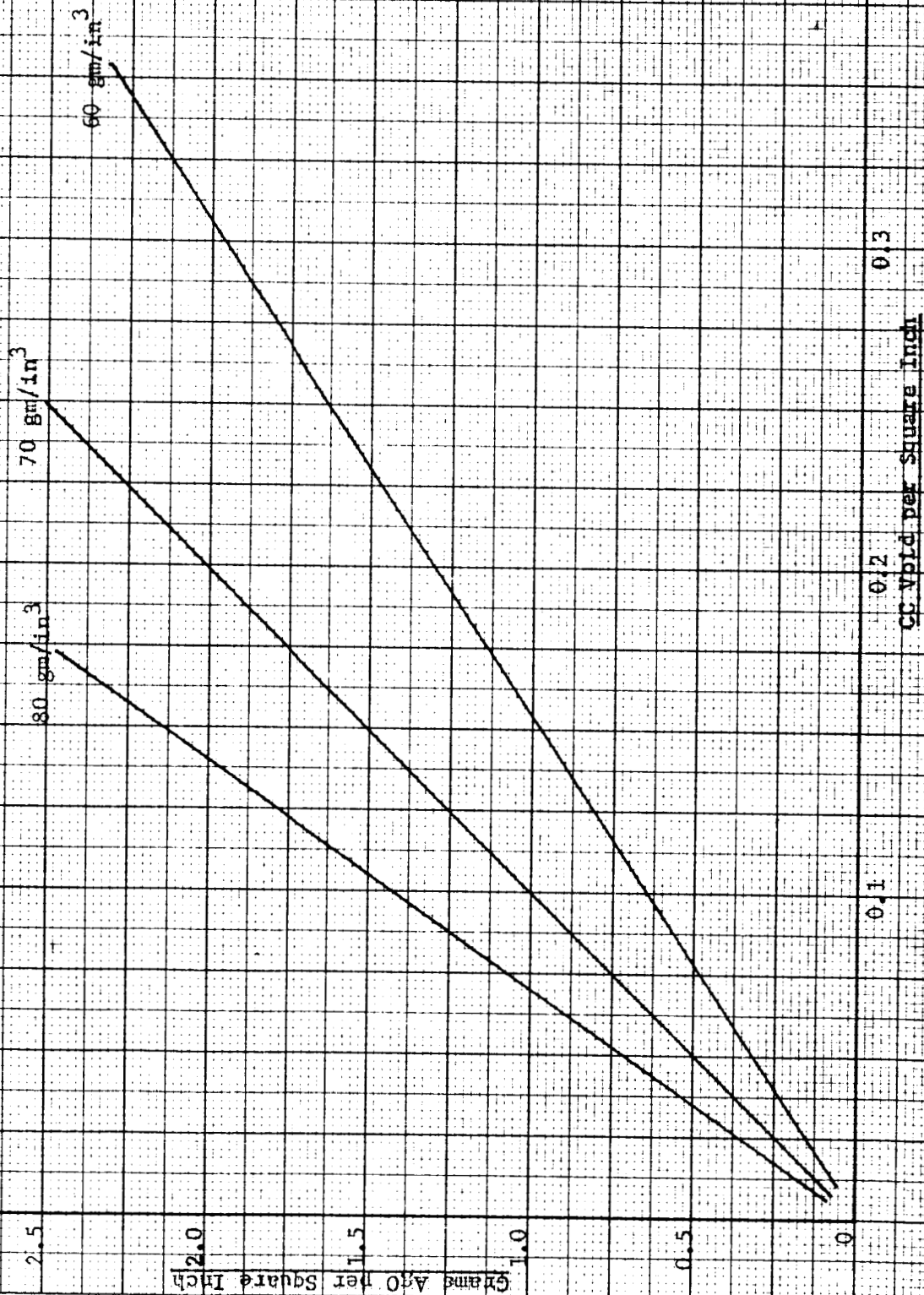


FIGURE NO. 12
THICKNESS OF THE ZINC PLATE

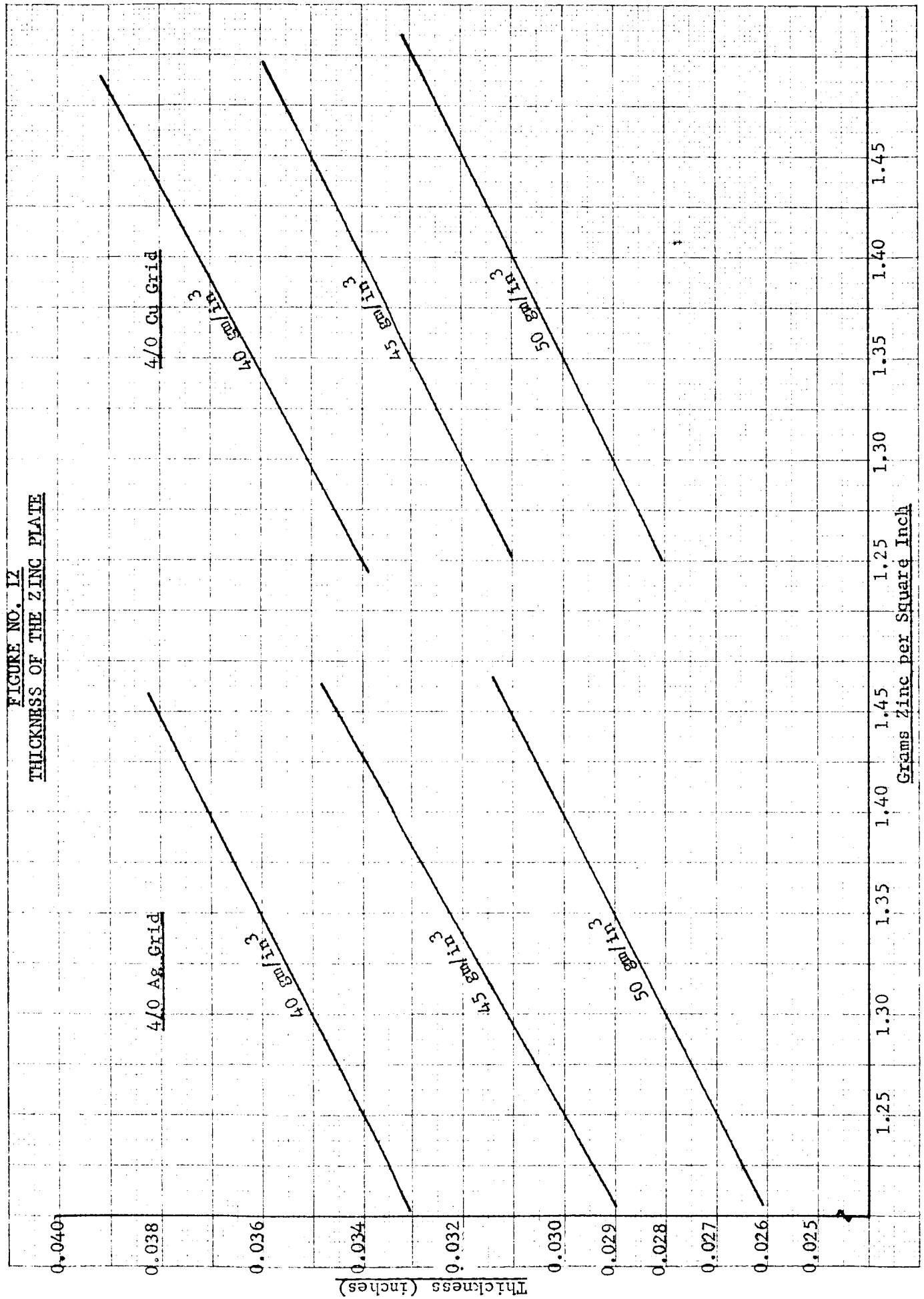
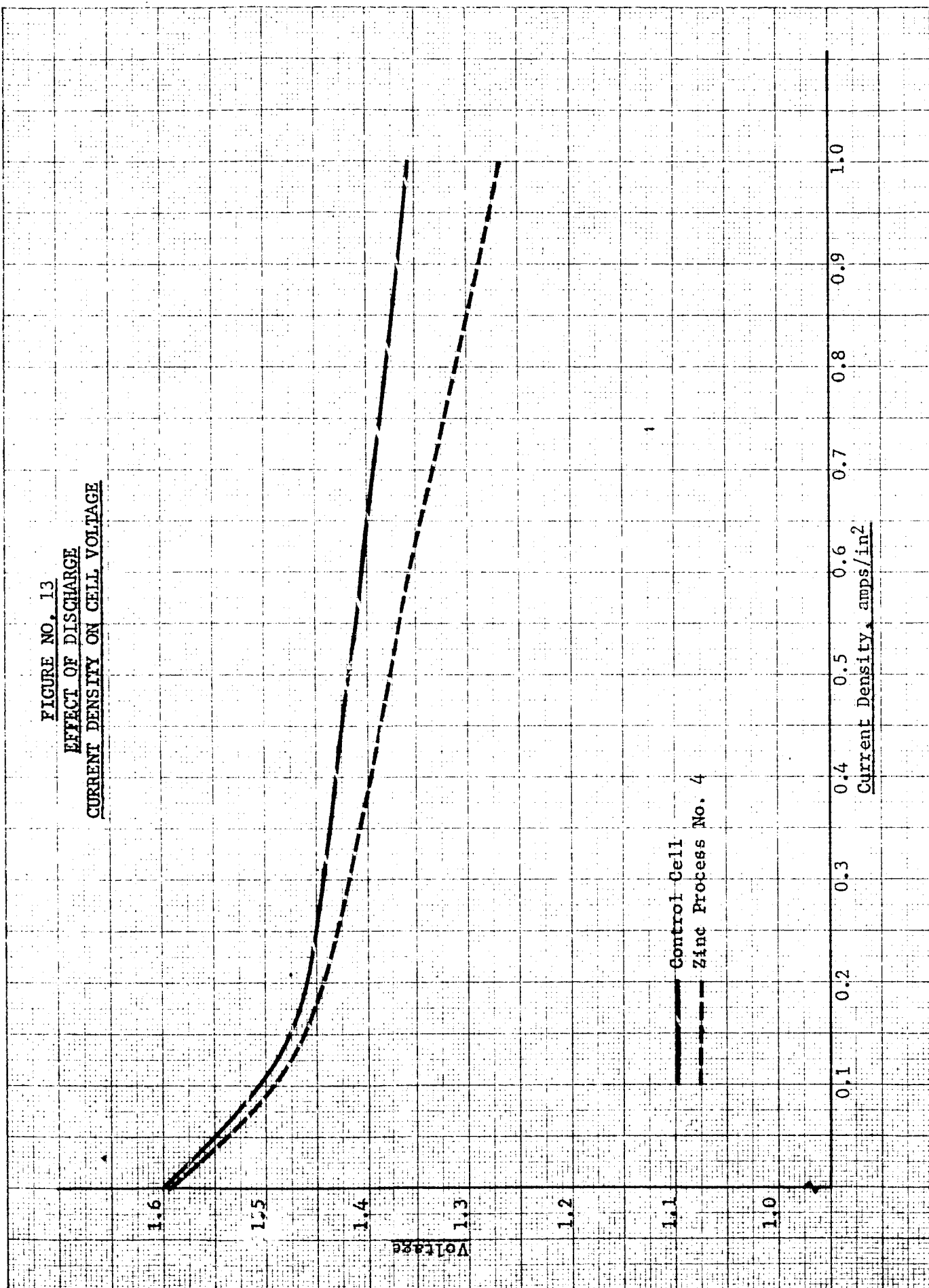


FIGURE NO. 13
EFFECT OF DISCHARGE
CURRENT DENSITY ON CELL VOLTAGE



3. Progress on Designed Experiment

The following is a discussion of the progress to date relative to the experiment designed to locate significant variables.

Discharges have been conducted on six of the cells, the variables of which have been outlined previously in this report. Data relative to these discharges are exhibited by Figure Nos. 14 through 19. Construction variables for each cell are itemized in conjunction with the respective discharge curve. All discharges in this series shall be conducted at a 30-ampere rate, which is equivalent to the 20 to 30-minute discharge rate. In addition, a 40-ampere surge of short duration shall be included in the discharge. The voltage exhibited during this surge is displayed also with the respective discharge curve. The voltage produced during this surge represents the maximum voltage to be anticipated during a 40-ampere discharge. It is not indicative of voltages produced in the opening minutes of 40-ampere discharges.

Discharges described in this report were conducted on individual cells so that heating effects on each cell would result from characteristics of that cell and not from its position within a "pack" of unlike cells. Also, this facilitated the rapid recording of cell voltages during the significant early portion of the discharges.

Mathematical treatment of data from the factorial experimental design series will have to await completion of all tests. It appears that there are three responses which might be studied in relation to cell discharges. They are: voltage, voltage control, and cell capacity. An arbitrary criterion for voltage control might be the percentage of the discharge time during which the cell voltage is greater than 0.9, the maximum discharge voltage exhibited.

One cell, No. 4-1 (the variables of which are listed in Figure No. 15), performed very well with respect to all three responses. It discharged for thirty minutes with a maximum of 1.45 volts which occurred after twelve minutes. In addition, during 28.5 minutes, or 95% of the discharge, the cell voltage was in excess of 1.30 volts. In contrast, Cell No. 2-1 discharged only 19 minutes of which 16, or 84%, were in excess of 1.30 volts. Efforts will be made to determine whether the characteristics of Cell No. 4-1 were reproducible and not random in nature.

Cells have been placed on +130° F stand and will remain at this temperature with two duplicate sets being removed and discharged at time intervals not yet determined. Others will be allowed to stand until failure.

It has proved to be impractical to produce and electroform in sufficient quantities, a special "pasted" negative material of the high quality required for a carefully controlled experiment. This material has not been previously used in primary cells. If no satisfactory substitute can be found for material of this formulation, data may be processed so that the absence of this level will not detract from the remainder of the experiment.

FIGURE NO. 14

Cell No. 2-1

40% KOH - No additive

Positive: 74 gm/in³ on 4/0 Ni

Negative: Metallic with 2% additive

50 gm/in³ on 4/0 Ag

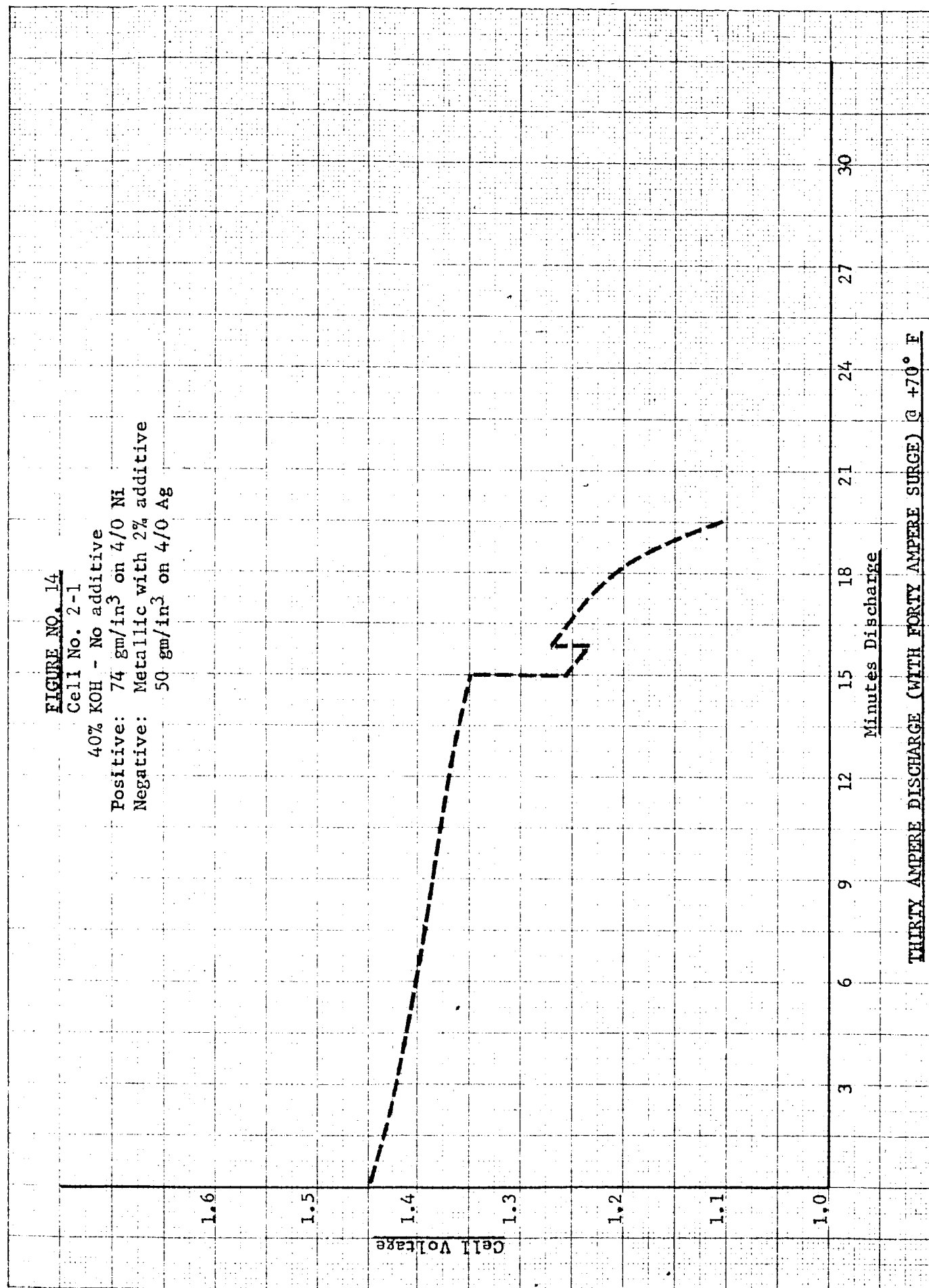


FIGURE NO. 15

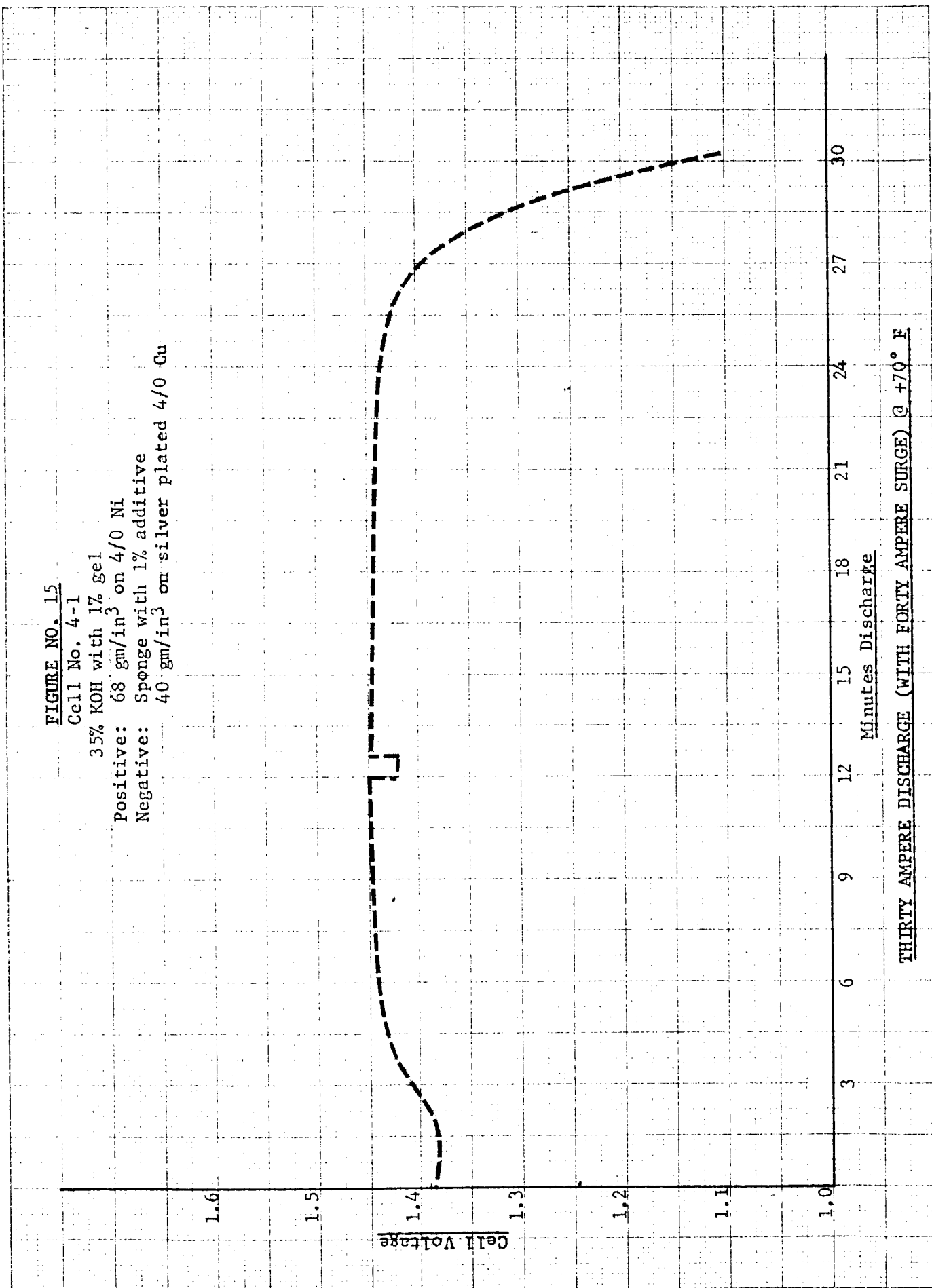
Cell No. 4-1

35% KOH with 1% gel

Positive: 68 gm/in³ on 4/0 Ni

Negative: Sponge with 1% additive

40 gm/in³ on silver plated 4/0 Cu



THIRTY AMPERE DISCHARGE (WITH FORTY AMPERE SURGE) @ +70° F

FIGURE NO. 16

Cell No. 7-1

35% KOH with MnO₂

Positive: 69 gm/in³ on 4/0 Ag

Negative: Metallic with 4% additive

40 gm/in³ on 4/0 Ag

1.6

1.5

1.4

1.3

1.2

1.1

1.0

Cell Voltage

30

27

24

21

18

15

12

9

6

3

Minutes Discharge

THIRTY AMPERE DISCHARGE (WITH FORTY AMPERE SURGE) @ +70° F

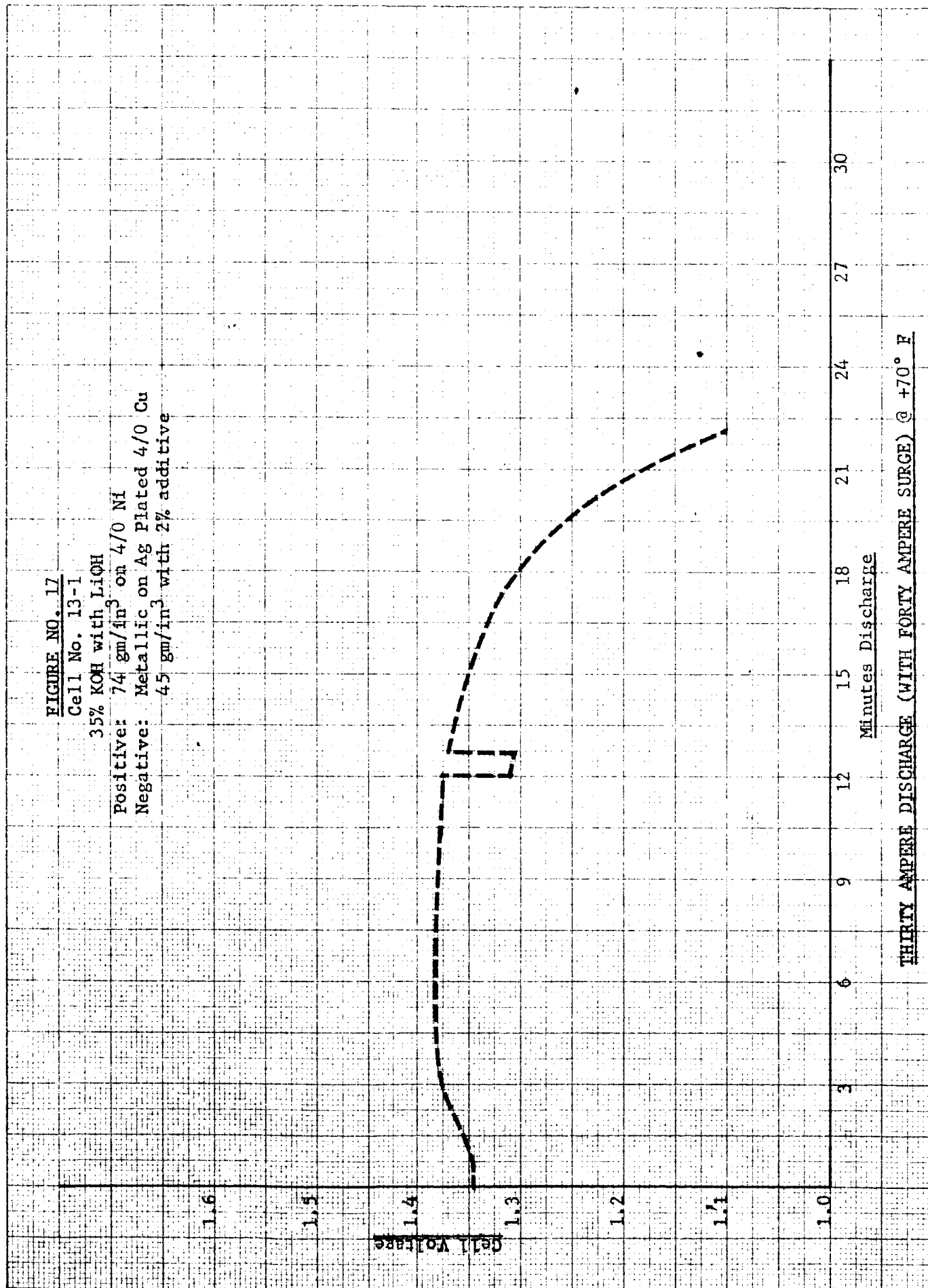
FIGURE NO. 17

Cell No. 13-1

35% KOH with LiOH

Positive: 74 gm/in³ on 4/0 Ni

Negative: Metallic on Ag Plated 4/0 Cu
45 gm/in³ with 2% additive



THIRTY AMPERE DISCHARGE (WITH FORTY AMPERE SURGE) @ +70° F

FIGURE NO. 18

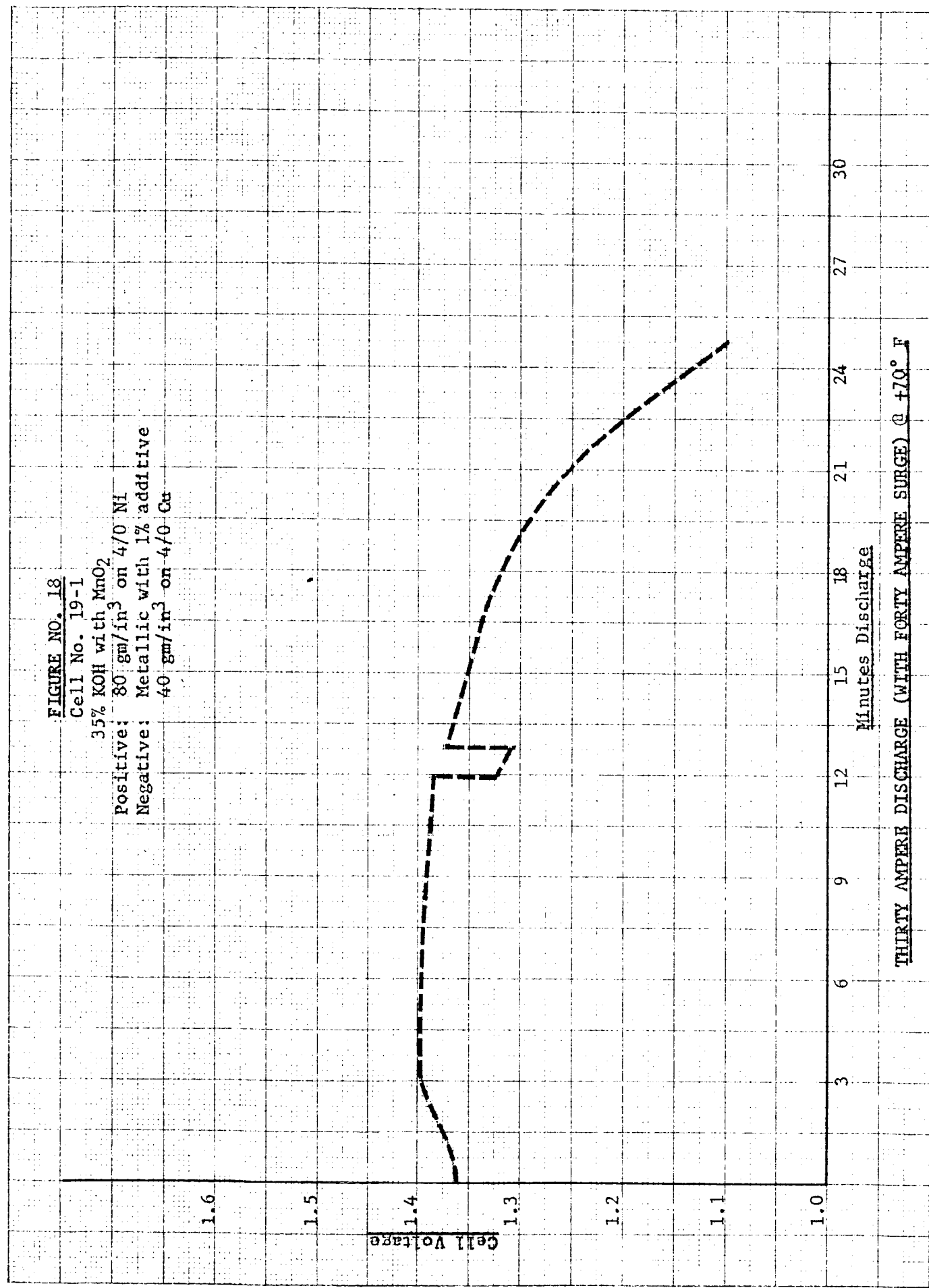
Cell No. 19-1

35% KOH with MnO₂

Positive: 80 gm/in³ on 4/0 Ni

Negative: Metallic with 1% additive

40 gm/in³ on 4/0 Cu



THIRTY AMPERE DISCHARGE (WITH FORTY AMPERE SURGE) @ +70° F

FIGURE NO. 19

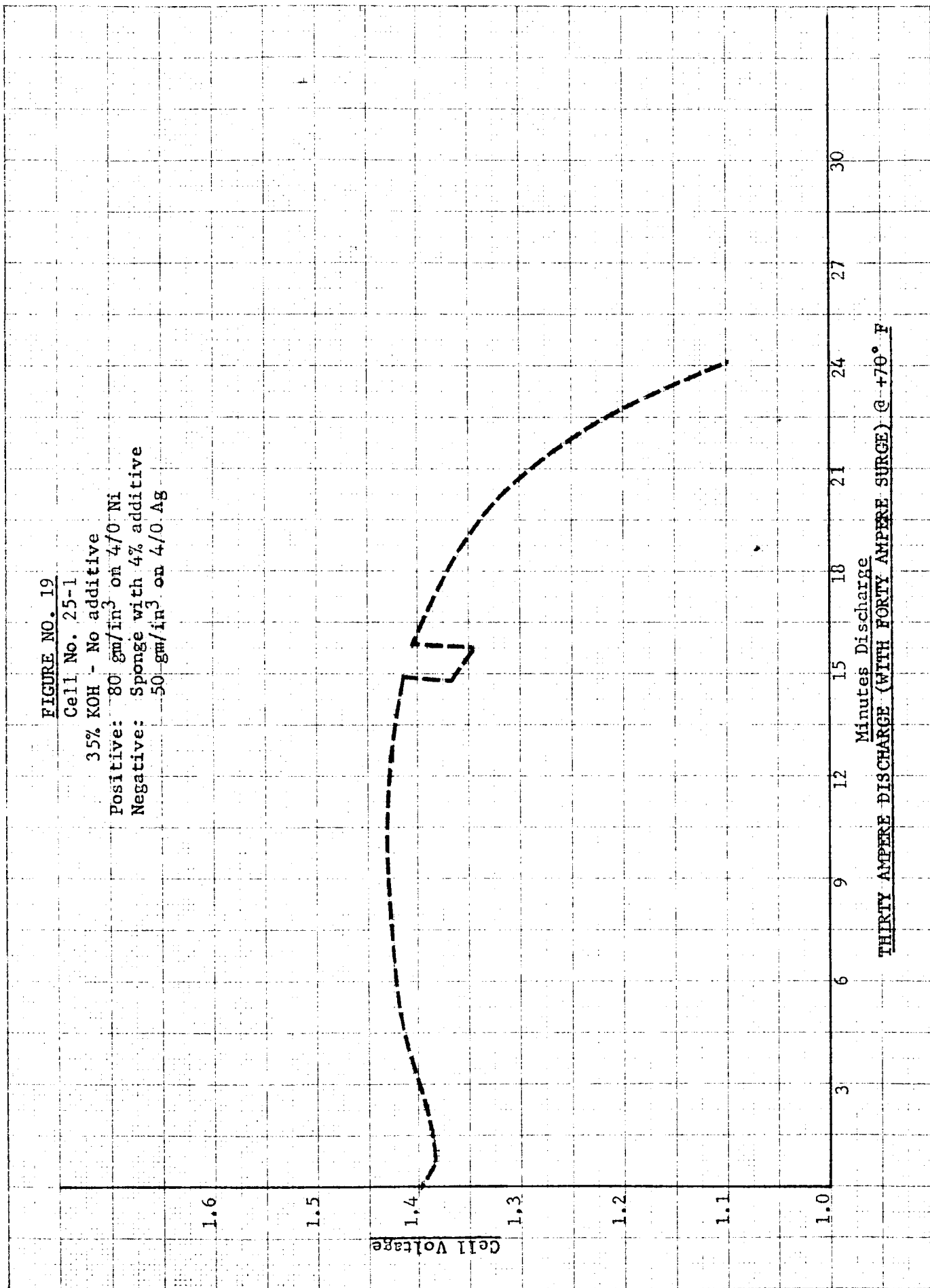
Cell No. 25-1

35% KOH - No additive

Positive: 80 gm/in³ on 4/0 Ni

Negative: Sponge with 4% additive

50 gm/in³ on 4/0 Ag



IV. CONCLUSIONS

1. Data indicate several separator combinations are capable of withstanding cell environments for periods of months at room temperature.
2. A high percentage of cells may be expected to retain useful capacity over a period of one to two months under laboratory conditions.
3. The control or "standard" cell is not the optimum cell for long stand applications.
4. A high percentage of carefully constructed cells using Cellophane, 9526 Nylon and R-35-D Viskon as separators may be expected to retain useful capacity after six to eight days at +130° F.
5. Data are insufficient to estimate battery reliability or determine optimum cell design.

V. PROGRAM FOR THE NEXT INTERVAL

1. Stand tests will continue at +130° F. This will include evaluations of several design factors.
2. Sufficient data should be gained to determine a desirable combination of cell design variables.
3. A series of cells of a pre-prototype design will be constructed and evaluated relative to cell performance prior to work under this contract.

VI. PERSONNEL

The following totals of man-hours have been expended during the contract period:

Engineering	- 694 hours
Technical	- <u>1530 hours</u>
TOTAL	- 2224 hours

BRH/bk

A P P E N D I X

MASTER SCHEDULE

PREPARED
APPROVED
APPROVED
APPROVED

PROGRAM	PRIMARY ZINC-SILVER OXIDE BATTERY
CONTRACT	NAS 8-5493
REFERENCE	FA-242

1 July 1963
P.O. DAT

1963		1964	
ACTIVITIES		CALENDAR	
		AFTER GO-AHEAD	
		JULY	
		I	
		II	
		III	
		IV	
		V	
		VI	
		VII	
		VIII	
		IX	
		X	
		XI	
		XII	
SEPARATOR STUDIES			
Open Types			
Closed Types			
Special Types			
ACTIVE MATERIAL FORMULATION			
Positive Material			
Negative Material			
PLATE CONSTRUCTION AND ELEMENT DESIGN			
Spongy vs. Metallic Zinc			
Dry Charged Spongy Zinc			
Grid Materials			
ELECTROLYTE			
Effect of Concentration			
Additives to Electrolyte			
CELL AND BATTERY CONSTRUCTION			
Electrolyte Retention			
Voltage Regulation			
Energy Density			
Thermal Characteristics			
Evaluation of State-of-Art			
REACTION MECHANISMS			
Theoretical Investigation			
Gassing Rate			
RETENTION EVALUATION			
PROGRESS REPORTS			

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